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RESEARCH MEMORANDUM

THE EFFECT OF MAGNESIUM PARTICLES OF VARIOUS EQUIVALENT DIAMETERS ON SOME PHYSICAL PROPERTIES OF PETROLATUM-STABILIZED MAGNESIUM-HYDROCARBON SLURRIES

By Joseph M. Lamberti

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

CLASSIFICATION CHANGED
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By NACA Res Abs.
Authority of RN-116 Date June 20, 1957
at 7-8-57

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RESEARCH MEMORANDUM

THE EFFECT OF MAGNESIUM PARTICLES OF VARIOUS EQUIVALENT DIAMETERS ON SOME PHYSICAL PROPERTIES OF PETROLATUM-STABILIZED MAGNESIUM-HYDROCARBON SLURRIES

By Joseph M. Lamberti

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SUMMARY

The effect of magnesium particles of various equivalent diameters on the apparent viscosity, sedimentation ratio, and redispersibility of petrolatum-stabilized magnesium - JP-4 slurries is described. The investigation was conducted in laboratory-scale apparatus. Powders of the following equivalent diameters were investigated: 2.8, 3.7, 7.2, 9.3, 12.0, and 14.8 microns. Data are also presented showing the effect of various magnesium concentrations on these physical properties.

INTRODUCTION

As a part of a general program to evaluate high-energy fuels for jet-propulsion systems, metals and metal-hydrocarbon slurries are currently being investigated at the NACA Lewis laboratory. As fuels, aluminum, magnesium, and boron have higher air specific impulse than the conventional hydrocarbon fuels (refs. 1 to 3). Boron, in addition, has higher fuel-specific-impulse characteristics than hydrocarbon fuels; it therefore would provide greater fuel economy and thus longer flight range (refs. 1 and 4).

In experimental investigations of ram-jet combustors, the slurry form of fuel has an advantage over solid fuel in that fuel flow is easily regulated. The preliminary combustion research on magnesium, aluminum, and boron, either as solid fuel beds or as slurries, is summarized in reference 1.

Since magnesium is a relatively inexpensive material and has a greater heat of combustion per pound of air than aluminum, the experimental investigation of magnesium slurries has progressed farther than those of either aluminum or boron. The preparations and physical properties of some magnesium slurries are described in references 5 to 7.

Heretofore, magnesium slurries have been prepared in which only the concentration of the metal or the type of carrier fuel was varied. The particle size of metal used in these preparations has been described as "fine" (approximately 66 microns), "ball-milled" (approximately 45 microns), and "superfine" (approximately 24 microns). There has been no detailed study of the effect of magnesium particle size on the physical properties of a slurry.

In the combustion studies of magnesium - fuel-oil slurries conducted in a $1\frac{7}{8}$-inch-diameter burner, 13- and 2-micron powders were used. It was noted that the 2-micron slurries had much higher blow-out velocities than did the 13-micron slurries (ref. 6).

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Inasmuch as the particle size affected the physical as well as the combustion properties of the slurry, a detailed systematic investigation was undertaken to determine the effect of varying particle size on the physical properties of the slurries.

In the present investigation of petrolatum-stabilized magnesium - JP-4 slurries, which was conducted on a laboratory scale, the following factors were considered:

(1) The particle size distribution of several magnesium powders with different average equivalent particle diameters

(2) The effect of the equivalent diameter of particles and the concentration of magnesium on apparent viscosity, sedimentation ratio, and redispersibility

(3) The effect of different petrolatums, purchased at different times under identical specifications, on the apparent viscosity

(4) The analysis of experimental error and reproducibility of results

DESCRIPTION OF POWDER AND CARRIER FUEL

The magnesium used in the preparation of the slurries described herein was fractionated into various particle size ranges by means of the Federal Laboratory Air Classifying Unit B. Particle size of each individual fraction obtained from this process was determined by the Roller analyzer. The method of the Roller analyzer, based on Stoke's Law, is accurate for spherical particles. Although the powder in these investigations was produced by an atomization process, not all of the particles were spherical; therefore, the particle size distribution is expressed in terms of an "equivalent spherical diameter."

The average particle diameter of each fraction obtained from the Federal Laboratory Air Classifying Unit B was determined by the Fisher Sub-Sieve Sizer. The number (average particle size) as obtained by this apparatus is six times the volume-surface ratio of the magnesium particles in that particular sample.

Throughout this report, the number as obtained by the Fisher Sub-Sieve Sizer for each batch of powder will be used to denote the equivalent diameter of the magnesium powder, and the phrase "average particle size of magnesium" will indicate "diameter of equivalent spherical particle of magnesium".

The data on the particle size distribution of the various magnesium powders are plotted in the form of bar graphs in figure 1. There is a correlation between the particle size distribution according to the Roller analysis and the equivalent particle diameter of each batch as determined by the Fisher Sub-Sieve Sizer except for the 14.8-micron powder. In this case, the Roller analysis indicated that 69 percent of the powder was in the range 17 to 40 microns and only 19 percent in the range 8.5 to 17.0 microns.

Photomicrographs of the various particle sizes of magnesium used in the preparation of slurries described in this report were taken with a metalloscope and are presented in figure 2.

The specifications and analysis of the MIL-F-5624A, grade JP-4 fuel used in this investigation are listed in table I. The petrolatum used in the determinations of sedimentation ratio and redispersibility is designated as petrolatum A. Those petrolatums used in the determinations of apparent viscosity were designated as petrolatums A and B. The physical properties of these materials are listed in table I.

PROCEDURE

Preparation of Slurries

The method of preparation of each slurry reported in this paper is essentially the one adopted in reference 7. Since the concentration of magnesium varied, the final ratio of petrolatum to JP-4 in each slurry was kept constant at 2:3 by weight.

Four stock solutions of petrolatum - JP-4, of 1800 grams total weight each, were prepared to make the four groups of slurries containing 30, 40, 50, and 60 percent by weight of magnesium. A typical stock solution was prepared by placing the correct amounts of petrolatum and JP-4 in a 1-gallon round tin-plated can and heating the mixture at a temperature of 140° F for 2 hours with vigorous stirring. Any loss of weight due to evaporation of the JP-4 was made up by the addition of more JP-4. The solution was then allowed to cool to room temperature.

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Individual slurries were prepared in quantities of approximately 750 grams each. In a typical preparation, the correct amount of magnesium of a particular particle size was placed in a 1-quart round tin-plated can and wetted thoroughly with 150 grams of JP-4. The necessary amount of the particular stock solution of petrolatum - JP-4 then was added to keep the individual slurry to the required proportions. For example, 750 grams of the slurry, 2.8-micron powder, 30 percent by weight of magnesium, was prepared in the following manner: 225 grams of 2.8-micron powder was placed in a tared can and wetted thoroughly with 150 grams of JP-4, followed by the addition of 375 grams of a stock solution of petrolatum (56 percent by weight) and JP-4 (44 percent by weight). The final composition of the resulting slurry was 225 grams of magnesium (30 percent), 315 grams of JP-4 (42 percent), and 210 grams of petrolatum (28 percent). The final ratio of petrolatum to JP-4 was 2:3 by weight. The resulting mixture was heated at a temperature of 140° F for 30 minutes with vigorous stirring. Any loss of weight of the mixture due to evaporation of the JP-4 was made up by the addition of more JP-4. The slurry was then allowed to remain overnight in a water bath at a temperature of 86° F.

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The slurries were aged in sealed containers for at least 2 weeks at room temperature prior to the determination of physical constants.

The following slurries containing petrolatum A were prepared:

Magnesium, percent by weight	Average particle size, microns					
	2.8	3.7	7.2	9.3	12.0	14.8
30	*	*	*	*	*	*
40	*	*	*	*	*	*
50	*	+	*	*	*	*
60	*	*	*	*	*	*

*Slurry prepared.
+Slurry not prepared, magnesium powder unavailable.

Slurries were also prepared from petrolatum B and JP-4 to determine the behavior of slurries prepared from a petrolatum of slightly different properties. These slurries had the following compositions: for the 7.2-micron powder, 30 percent by weight of magnesium; 7.2-micron, 40 percent; 9.3-micron, 50 percent; and 14.8-micron, 60 percent.

Determination of Physical Properties

Viscosity. - Viscosities were determined at 86° ±1.0° F with a model LVF Brookfield Synchro-lectric viscometer. The measurements were taken

with a spindle speed of 12 rpm, 30 seconds after the spindle had started to rotate. Several successive determinations were made until consistent results were obtained. Maximum variation in results did not exceed 10 percent for any slurry.

Sedimentation ratio. - Approximately 50 milliliters of slurry, immediately after vigorous stirring to give a uniform sample, was placed in a 2- by 22.5-centimeter graduated glass cylinder. The cylinder was sealed and taped to prevent evaporation and was then stored in a water bath at a temperature of 86° F for 4 weeks.

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Observations were made upon the stored slurry during the 4-week period. The depth of the clear supernatant liquid layer which formed above the metal-liquid layer was measured at frequent intervals during the storage period for the determination of the settling rate. The degree of settling of the slurry was expressed in terms of a sedimentation ratio (ref. 7) calculated from the following expression:

$$\text{Sedimentation ratio} = \frac{\text{Concentration of Mg in original slurry}}{\text{Concentration of Mg in settled portion of the slurry}}$$

Specifically, the sedimentation ratio was calculated from the following formula:

$$\text{Sedimentation ratio} = \frac{V_s d_s - \left(V_s - V_s \frac{h_t}{h_0}\right) d_m}{V_s d_s}$$

This equation may be simplified to:

$$\text{Sedimentation ratio} = \frac{d_s - d_m + d_m \frac{h_t}{h_0}}{d_s}$$

where

V_s volume of original slurry

d_s calculated density of original slurry

d_m calculated density of medium

h_0 total height of slurry when first placed in the graduated glass cylinder

h_t height of metal-liquid phase of the slurry in graduated glass cylinder at time t

[redacted]

The densities of the slurry and the medium may be calculated from the equations:

$$\frac{1}{\text{Density of slurry}} = \frac{\text{Weight fraction of magnesium}}{\text{Density of magnesium}} + \frac{\text{Weight fraction of medium}}{\text{Density of medium}}$$

$$\frac{1}{\text{Density of medium}} = \frac{\text{Weight fraction of petrolatum}}{\text{Density of petrolatum}} + \frac{\text{Weight fraction of JP-4}}{\text{Density of JP-4}}$$

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Redispersibility. - The extent to which a slurry could be redispersed was determined by an empirical method. When settling tests were concluded at the end of 4 weeks, the same sample in the graduated glass cylinder was placed horizontally in a shaker, shown in figure 3, and shaken for 5 minutes. The shaker, built at this laboratory and described in reference 7, oscillated 172 times per minute through an arc of 59°. After the shaking process, the glass cylinder was removed from the shaker and placed at a 45° angle from the horizontal while the fluid portion of the contents was carefully poured out. The cylinder, at this angle, was permitted to drain for one-half minute. The height of the portion of the slurry which did not pour out was noted. The percent redispersibility was calculated from the expression:

$$\text{Percent redispersibility} = \frac{h_t - h_l}{h_0} \times 100$$

where

h_0 total height of slurry when first placed in graduated glass cylinder

h_t height of metal-liquid phase of slurry at time t

h_l height of that portion of slurry which did not pour out after the shaking process

DISCUSSION OF RESULTS

Viscosity. - The viscosities of the slurries are listed in table II. The effect of the average particle size of metal on viscosity of magnesium slurries containing 30, 40, 50, and 60 percent by weight of metal and stabilized with petrolatum is shown in figure 4. For convenience, the viscosities in figure 4 were plotted on a logarithmic scale. Since, these slurries are non-Newtonian, the viscosity data are expressed in terms of "apparent viscosity", that is, the viscosities the slurries would have if they were Newtonian (ref. 8).

As the average particle size of metal is increased from 2.8 to 14.8 microns in slurries containing the same concentration of magnesium, minimums are observed in the apparent viscosities. For the slurries containing 30 percent magnesium the minimum value for the apparent viscosity appears for the particle size of 9.3 microns; for the 40 percent, 7.2 microns; for the 50 percent, 9.3 microns; and for the 60 percent, 12.0 microns. Although a minimum does appear for the 60-percent-magnesium slurries investigated, particle size has little or no effect on the apparent viscosity of the slurry in the range from 7.2 to 14.8 microns.

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There is very little difference between the apparent viscosities of the 2.8-micron and the 14.8-micron slurry of the 30-percent concentration of magnesium. However, for the 60-percent magnesium concentration, the difference in the apparent viscosity is great. The apparent viscosity of the 2.8-micron slurry is three and one-half times greater than that of the 14.8-micron slurry.

Figure 4 shows also that, in the 30- to 60-percent-magnesium concentration range, the apparent viscosity of any group of slurries containing the same average size magnesium particles increases as the concentration of the magnesium is increased. The slight decrease which is noted in the 2.8-micron slurry as the concentration of magnesium is increased from 50 to 60 percent is due probably to experimental error.

Sedimentation ratio. - The effect of time on the sedimentation ratios of petrolatum-stabilized magnesium - JP-4 slurries is shown in figure 5. The determination of the sedimentation ratio is an attempt to measure quantitatively the degree of settling of a slurry (ref. 7). A high sedimentation ratio indicates a small amount of settling for any given slurry.

The rate of settling for the slurries was rapid in the first 5 days; then the slope of the curve gradually diminished until a plateau was reached at about 18 days.

At the end of 30 days, the slurries, according to particle size, had sedimentation ratios in the following order:

Magnesium, percent by weight	Average particle size, microns					
30	2.8	>3.7	>12.0	>9.3	>14.8	>7.2
40	2.8	>7.2	>12.0	>9.3	> 3.7	>14.8
50	2.8	>7.2	>12.0	>14.8	> 9.3	(a)
60	2.8	> 3.7	>7.2	> 9.3	>14.8	>12.0

[a]3.7-micron slurry not prepared; magnesium powder unavailable.

[redacted]

The results shown in this table indicate that particle size generally influences the sedimentation ratio of slurries: the smaller the particle size, the greater the sedimentation ratio or the lower the amount of settling. Of all the slurries studied in this investigation, the 2.8-micron slurry had the greatest sedimentation ratio, regardless of the concentration of magnesium in the slurry.

The effect of particle size of magnesium on the sedimentation ratio at the end of 2 and 14 days is shown in figure 6, a cross-plot of the data from figure 5. Minimums are observed in these sedimentation ratios when plotted as a function of particle size. A comparison of these minimums with those of the apparent viscosities, shown in table III, indicates that both minimums generally occur in the same area of particle size of magnesium except for the 30-percent concentration of magnesium. In this case, the sedimentation ratio minimums occur at 3.7 microns, at the end of 2 days, and at 7.2 microns, at the end of 14 days. In contrast, the viscosity minimum is observed at 9.3 microns. It is noted also in table III that the sedimentation ratio minimum for the 40-percent concentration of magnesium, at the end of 14 days, occurs at 9.3 microns instead at 7.2 microns. The difference between the sedimentation ratios for these two slurries is probably within experimental error, however.

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Redispersibility. - The redispersibility (ratio of the amount of slurry that poured out after the shaking process to the total amount of slurry placed in the graduate) was determined for each slurry following the determination of the sedimentation ratio at 30 days.

The effect of particle size of magnesium on redispersibility is shown in figure 7. An increase in particle size produces an increase in redispersibility for slurries of 50- and 60-percent concentrations of magnesium. However, for the 30- and 40-percent concentrations, an increase in particle size causes a decrease in the percent redispersibility except in two instances: 14.8-micron, 30-percent magnesium and 7.3-micron, 40-percent magnesium, where an increase is observed.

The effect of concentration of magnesium on the redispersibility of the slurries investigated in this report is shown in figure 8. In the smaller average particle sizes (2.8, 3.7, and 7.2 microns), the percent redispersibility decreases rather sharply with increase in concentration of magnesium in the order: 30 > 40 > 50 > 60 percent. However, in the larger particle size range (9.3, 12.0, and 14.8 microns), the decrease in percent redispersibility is slight, except for the 14.8-micron, 40-percent, and in the 9.3-micron, 60-percent magnesium slurries.

Although the 2.8-micron slurry had the greatest sedimentation ratio (least amount of settling) of the slurries investigated, it had the greatest decrease in percent redispersibility with an increase in the concentration of magnesium in a given slurry.

EFFECT OF DIFFERENT PETROLATUMS

Two different batches of petrolatums, designated as petrolatum A and petrolatum B, were purchased at different times under the same specifications, and the physical properties of the materials are presented in table I. The following slurries were selected to determine the effect of different petrolatums on the apparent viscosity and sedimentation ratio: 7.2-micron slurry, 30 percent by weight of magnesium; 7.2-micron, 40 percent; 9.3-micron, 50 percent; and 14.8-micron, 60 percent. These slurries were prepared and aged as described previously. In the first set of slurries, petrolatum A was used; in the second set, petrolatum B.

Viscosity. - Viscosities of the eight stock solutions, with no magnesium added and with either of the petrolatums with JP-4, were determined under the same experimental conditions. The results are summarized in table IV.

The data in this table indicate that the type of petrolatum affects the measured viscosity of the stock solutions. Viscosity determinations of the selected slurries, with either petrolatum A or petrolatum B and JP-4, were determined on 100 milliliters of material in 100-milliliter pyrex beakers. The results (table V) indicate that slurries of the same composition, but with two different samples of petrolatum purchased under the same specifications, had different viscosities when the determinations were made with the same quantity of material in the same type of container.

The percent difference in the viscosity of the slurries prepared from different petrolatums (table V) is smaller than the percent difference in the viscosity of the stock solutions of the different petrolatums with JP-4 (table IV). This may have been caused by the smaller proportion of petrolatum in the slurry than in the stock solution.

It was observed in the determination of viscosity measurements that viscosity values for slurries of the same composition containing the same type of petrolatum were reproduced within 10 percent when determinations were made with the same quantities of slurry in the same type of container. However, the viscosity values varied more than 10 percent when 20 percent of the slurry was removed from the container or when a different size container was used. A series of tests was conducted to determine the effect of container size, as well as other factors, that may influence viscosity values when determined with a Brookfield viscometer. The data are presented in the appendix.

Sedimentation ratio. - The two sets of determinations of the sedimentation ratios of each of the four selected slurries prepared with petrolatum A are plotted in figure 9. The sedimentation ratios taken a month later using another sample of the same slurry were within 10 percent of the original determinations.

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Redispersibility. - The 2.8-micron slurry of various concentrations of magnesium was used to determine the reproducibility of the redispersibility of a slurry. The second determination was made a month following the first determination of the same slurry. The results are summarized below:

Magnesium, percent by weight	Redispersibility, percent		Difference[c], percent
	(a)	(b)	
30	91.7	91.5	0.22
40	79.7	77.3	3.01
50	30.8	30.3	1.62
60	0	0	0

[a]First determination.
[b]Second determination, a month later.
[c]Based on the first determination.

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SUMMARY OF RESULTS

The effect of magnesium particles of various equivalent diameters (2.8, 3.7, 7.2, 9.3, 12.0, 14.8 microns) on the apparent viscosity, sedimentation ratio, and redispersibility of petrolatum-stabilized magnesium - JP-4 slurries was studied. The following results were obtained:

1. As the average particle size of metal was increased from 2.8 to 14.8 microns in slurries containing the same concentration of magnesium, minimums were observed in the apparent viscosity - magnesium concentration curves.

2. The sedimentation ratio increased as the concentration of magnesium in a slurry was increased. Particle size influenced the settling of magnesium slurries as follows: the smaller the particle size of metal, the smaller the amount of settling.

3. The degree of redispersibility of a slurry decreased as the concentration of magnesium within the slurry was increased. Particle size of magnesium also affected the redispersibility of a slurry as follows: increasing particle size of magnesium produced an increased percentage of redispersibility for slurries of 50- and 60-percent concentration of magnesium. However, with slurries containing 30 and 40 percent magnesium, an increase in the magnesium particle size produced a decrease in the percent redispersibility except in two instances: 14.8-micron, 30-percent magnesium slurry; and 7.3-micron, 40-percent magnesium slurry. In these cases, increases were observed.

4. Although the 2.8-micron slurry had the greatest sedimentation ratio (least amount of settling), it showed the greatest decrease in the percent redispersibility as the concentration of magnesium in a given slurry was increased.

In the analysis of experimental error and reproducibility of values of apparent viscosity, sedimentation ratio, and redispersibility of the slurries that were investigated, the following evaluations were made:

1. The experimental viscosity, sedimentation ratio, and percent redispersibility values could be reproduced for the same slurry when determined under identical experimental conditions.

2. The apparent viscosity as determined by the Brookfield instrument was exceedingly dependent, for the slurry investigated, on experimental variables such as the size of the vessel containing the slurry sample, spindle speed, spindle position, and time of rotation.

3. The apparent viscosity value was dependent on the particular batch of petrolatum used.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, January 26, 1954

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APPENDIX - EVALUATION OF VISCOSITY MEASUREMENTS

One of the important properties for the evaluation of a metal-hydrocarbon slurry as a potential high-energy fuel is viscosity. Metal-hydrocarbon slurries are non-Newtonian in behavior and consequently such factors as thixotropy, plasticity, pseudoplasticity, and dilatancy may affect viscosity measurements of the slurries. In many cases, viscosity values are not only dependent on the type of viscometer used but also on the specific technique employed. In comparing or correlating viscosities of different slurries, it is important that the factors involved in the measurement of the viscosities be taken also into consideration. 3131

Viscosity evaluations described herein are limited to those determined at 86° ±1.0° F with a Brookfield viscometer (ref. 7). The number 3 spindle of the instrument was used in all the determinations, and the level of the slurry in the container was always maintained at the groove mark of the spindle. The 7.2-micron slurry, 40 percent by weight of magnesium, was used in all of the tests. Three additional slurries were used to study the effect of container size on the viscosity of a slurry: 7.2-micron slurry, 30 percent by weight of magnesium; 9.3-micron, 50 percent; and 14.8-micron, 60 percent. All these slurries containing petrolatum A were prepared and aged as described in the main body of the report.

Series of tests were conducted to determine some of the factors that may be important in the determination of viscosity of a metal-hydrocarbon slurry with the Brookfield viscometer. It is not implied that all the factors discussed herein affect viscosity measurements for any other material or for any other type of slurry to the same degree that was found in the investigation of this specific type of slurry. No attempt is made to analyze the physical character of the slurry in terms of the viscosity data obtained. Since a metal-hydrocarbon slurry is non-Newtonian, the viscosity data are expressed in terms of "apparent viscosity", that is, the viscosity the slurry would have if it were Newtonian (ref. 8). For convenience, several of the graphs have been plotted on semilogarithmic coordinates.

The tests conducted to evaluate some of the important factors in the determination of viscosity for metal-hydrocarbon slurries with a Brookfield viscometer are described in the following paragraphs.

Container size. - Three sets of viscosity measurements were made of the four selected slurries described: (1) 750 grams of slurry in a 1-quart round tin-plated container, (2) the same slurry in the same container but with 150 grams of slurry removed, and (3) 100 milliliters of the same slurry poured in a 100-milliliter pyrex beaker. The measurements were taken with a spindle speed of 12 rpm, 30 seconds after the spindle started to rotate. The position of the spindle and the baffle of the instrument was approximately in the center of the container for each determination. The results are tabulated in table VI.

The viscosity values, as shown in table VI, for slurries of the same composition were reproduced within 10 percent when determinations were made with the same quantities of slurry in the same type of container. It is observed that the viscosity values for each slurry varied more than 10 percent when 20 percent of the slurry was removed from the container. When a 100 milliliter beaker was used instead of a 1-quart can, the viscosity value measured with the Brookfield apparatus was greatly different.

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In order to study the effect of container size on the viscosity of a slurry in more detail, viscosity measurements were made of the 7.2-micron slurry, 40 percent by weight of magnesium in three different size containers: a 100-milliliter glass beaker (5 cm I.D.); a 400-milliliter glass beaker (8 cm I.D.); and a round, tin-plated quart can (10.4 cm I.D.). Determinations were made with the number 3 spindle at spindle speeds of 6, 12, 30, and 60 rpm. Readings were taken 30, 60, 90, and 120 seconds after the spindle started to rotate. The data are tabulated in table VII.

The effect of container size on the apparent viscosity of the slurry is shown in figure 10. Since there were only three points through which to draw a curve, the shape of the curve may not be accurate, but the effect of container size on the viscosity of the slurry is apparent. In each instance, for spindle speeds of 6, 12, and 30 rpm at various time intervals after the spindle began to rotate, the viscosities in the 100- and 400-milliliter beakers were higher than those in the quart-can. This seems to indicate that a greater drag was produced by the walls of the narrower containers on the rotating spindle, resulting in higher viscosity values. It is noted that the viscosity of the slurry in the 400-milliliter beaker was higher than that in the 100-milliliter beaker, but no explanation can be offered at this time.

Figure 10 shows also that for spindle speeds of 60 rpm, at 30, 60, 90, and 120 seconds after the spindle began to rotate, the viscosity increases as the size of container increases. This discrepancy may be caused by the low starting torque of the motor of the Brookfield viscometer. Viscosity measurements were made probably before the motor had an opportunity to reach its synchronous speed.

Time. - Measurements were made on approximately 600 grams of slurry in a round quart can (10.4 cm I.D.). The number 3 spindle of the viscometer was used at a spindle speed of 12 rpm, and readings were taken 30 seconds after the spindle started to rotate. After each determination, the instrument was turned off. The can was not removed from the instrument and the slurry was not restirred. Each successive determination was made 5 minutes after the preceding one without changing position of the spindle and baffle within the container.

The effect of time on apparent viscosity of this particular slurry is shown in figure 11. This figure shows that using the same spindle and spindle speed and keeping the relative position of the spindle and baffle within the container constant made possible apparent viscosity values which could be reproduced within 10 percent of the original determination for at least a 30-minute period without restirring the slurry.

Position of baffle and spindle. - The series of tests just described was repeated except that three different positions of the baffle and spindle within the slurry were used. The results, shown in figure 12, indicate that when the same spindle and spindle speed are used, viscosity values can be reproduced within 10 percent provided that the position of the baffle and spindle within the slurry did not change. Changes in the position of the baffle and spindle, shown diagrammatically in figure 12, caused variations from 18 to 48 percent of the original set of determinations. When the position of the baffle and spindle were reset in the original position, the results varied within 1 percent of the original set of viscosity determinations. As the baffle and spindle were moved gradually nearer to the wall of the container, a greater drag was probably produced upon the rotating spindle resulting in a higher viscosity value.

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Time interval after the spindle started to rotate. - The effect of time after the spindle started to rotate on the apparent viscosity of a slurry in three different size containers is shown in figure 13. The apparent viscosity of the slurry in all three containers decreased as time (in the range of 30 to 120 sec) increased, regardless of the spindle speed (in the range of 6 to 60 rpm). The shape of the curves may denote a gradual breakdown of the internal physical configuration of the slurry as time increases. The effect is very slight for the larger container (quart can), indicating that any additional effect of the drag created by the walls of the container on the rotating spindle in hastening the breakdown of the slurry is less than in the smaller containers (100- and 400-ml beakers).

It is noted that the apparent viscosity of the slurry in the quart can decreases more rapidly as time increases for the spindle speed of 60 rpm than for spindle speeds of 6, 12, and 30 rpm. This discrepancy, as stated previously, may be caused by the inaccurate viscosity measurements made before the motor of the instrument had an opportunity to reach its synchronous speed.

Spindle speed. - The effect of spindle speed on the apparent viscosity of a slurry in three different size containers is shown in figure 14. The curves indicate that as spindle speed is increased, the apparent viscosity of the slurry in each container is decreased. The rate of decrease in the apparent viscosity is caused by the higher rates of shear produced by the greater spindle speeds. The exception is noted for the slurry in the quart can at the spindle speed of 60 rpm, which, again, can be explained by the fact that the motor of the Brookfield

viscometer has a low starting torque. Measurements were obtained before the motor had an opportunity to reach its synchronous speed at this spindle speed.

Figure 14 shows also that at any specific spindle speed, the apparent viscosity of the slurry decreases as the time interval after the spindle started to rotate is increased from 30 to 120 seconds, which again suggests a gradual breakdown of the internal configuration of the slurry as time increases.

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TABLE I. - SPECIFICATIONS AND ANALYSIS OF CARRIER FUEL

(a) MIL-F-5624A, grade JP-4 fuel.

	Specifications	Analysis
A.S.T.M. distillation method D 86-46, °F		---
Initial boiling point		140
Percent evaporated		---
5		199
10		222
20		248
30		268
40		286
50		300
60		325
70		348
80		382
90		427
Final boiling point	550(max.)	488
Residue, percent	1.5(max.)	1.0
Loss, percent	1.5(max.)	0
Specific gravity, 60°/60° F		0.768
Reid vapor pressure, lb/sq in.	2 to 3	2.5
Hydrogen-carbon ratio		0.169
Net heat of combustion, Btu/lb	18,400(min.)	[a]18,675

[a]Aniline point correlation.

(b) Petrolatums.

	Petrolatum	
	A	B
Melting point (Saybolt), °F	161	159
Penetration, A.S.T.M.	80	72
Viscosity (Saybolt) at 210° F, sec	101	91

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TABLE II. - BROOKFIELD APPARENT VISCOSITIES OF VARIOUS PETROLATUM-STABILIZED MAGNESIUM - JP-4 SLURRIES

Average particle size, microns	Brookfield apparent viscosity[a], centipoises			
	Magnesium concentration, percent by weight			
	30	40	50	60
2.8	870	1270	9900	8580
3.7	730	740	(a)	4240
7.2	600	[b]710	1400	2240
9.3	[b]590	820	[b]1100	2200
12.0	750	960	1720	[b]2100
14.8	900	980	1800	2250

[a]Temperature, 86° ±0.1° F.
[b]Minimum observed value.

TABLE III. - COMPARISON OF THE MINIMUMS OF BROOKFIELD APPARENT VISCOSITIES AND SEDIMENTATION RATIOS OF VARIOUS MAGNESIUM CONCENTRATIONS OF PETROLATUM-STABILIZED MAGNESIUM - JP-4 SLURRIES

Average particle size, microns	Magnesium, 30 percent			Magnesium, 40 percent		
	Viscosity, centipoises 86° ±1.0° F	Sedimentation ratio at end of 2 days	Sedimentation ratio at end of 14 days	Viscosity, centipoises 86° ±1.0° F	Sedimentation ratio at end of 2 days	Sedimentation ratio at end of 14 days
2.8	870	0.893	0.766	1270	1.000	0.876
3.7	730	[a].741	.708	740	.930	.780
7.2	600	.775	[b].678	[c]710	[a].848	.771
9.3	[c]590	.798	.700	820	.884	[b].766
12.0	750	.901	.717	960	.935	.775
14.8	900	.773	.693	980	.922	.770
	Magnesium, 50 percent			Magnesium, 60 percent		
2.8	9900	0.996	0.976	8580	1.000	0.982
3.7	(d)	(d)	(d)	4240	.986	.953
7.2	1440	.951	.899	2240	.974	.936
9.3	[c]1100	[a].901	[b].841	2200	.956	.921
12.0	1720	.942	.855	[c]2100	[a].938	[b].908
14.8	1800	.927	.853	2250	.968	.917

[a]Minimum observed in sedimentation ratio at the end of 2 days.
[b]Minimum observed in sedimentation ratio at end of 14 days.
[c]Minimum observed in viscosity.
[d]Slurry not prepared; magnesium powder unavailable.

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TABLE IV. - BROOKFIELD APPARENT VISCOSITIES OF VARIOUS PETROLATUM - JP-4 STOCK SOLUTIONS

Stock solution, percent by weight	Brookfield apparent viscosity[a], centipoises		Percent difference[b]
	Petrolatum A	Petrolatum B	
JP-4, 44; petrolatum, 56 (used in preparing 30-percent magnesium slurries	2250	6680	66.3
JP-4, 40; petrolatum, 60 (used in preparing 40-percent magnesium slurries)	5380	8200	34.4
JP-4, $33\frac{1}{3}$; petrolatum, $66\frac{2}{3}$ (used in preparing 50-percent magnesium slurries)	7730	15,800	51.1
JP-4, 20; petrolatum, 80 (used in preparing 60-percent magnesium slurries)	76,300	61,200	(c)

[a]Temperature, $86^{\circ} \pm 1.0^{\circ}$ F.
[b]Based on petrolatum B.
[c]Values unreliable; material too viscous to wet completely the spindle of the instrument.

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TABLE V. - BROOKFIELD APPARENT VISCOSITIES OF SELECTED SLURRIES CONTAINING DIFFERENT PETROLATUMS

Slurry		Brookfield apparent viscosity[a], centipoises		Percent difference[c]
Particle size, microns	Magnesium concentration, percent by weight	Petrolatum A[b]	Petrolatum B[b]	
7.2	30	940	1140	17.5
7.2	40	1450	1860	21.7
9.3	50	2650	3200	17.2
14.8	60	5610	5580	0.4

[a]Temperature, 86° ±1.0° F.

[b]Viscosity measurement taken on 100 ml of slurry in a 100-ml pyrex beaker.

[c]Based on petrolatum B.

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TABLE VI. - BROOKFIELD APPARENT VISCOSITIES OF SELECTED MAGNESIUM - JP-4 SLURRIES UNDER VARIOUS EXPERIMENTAL CONDITIONS

Slurry		Brookfield apparent viscosity[a], centipoises						
		750 g of slurry in round quart can			150 g of slurry removed from can		100 ml of slurry in 100-ml glass beaker	
Particle size, microns	Magnesium concentration, percent by weight	First determination	Second determination, 1 month later	Percent difference[b]	Reading	Percent difference[b]	Reading	Percent difference[c]
7.2	30	600	620	3.3	670	12	940	57
7.2	40	710	710	0	1030	69	1450	102
9.3	50	1100	1160	5.5	1550	41	2650	132
14.8	60	2250	2300	2.2	4100	82	5610	149

[a]Temperature, 86° ±1.0° F.

[b]Based on first determination.

[c]Based on first determination of 750 g of slurry.

TABLE VII. - BROOKFIELD APPARENT VISCOSITIES OF PETROLATUM-STABILIZED MAGNESIUM - JP-4 SLURRY CONTAINING 40 PERCENT MAGNESIUM OF 7.2-MICRON PARTICLE SIZE

Spindle speed[a], rpm	Brookfield apparent viscosity[b], centipoises											
	100-ml glass beaker (I.D., 5 cm)				400-ml glass beaker (I.D., 8 cm)				Round quart can (I.D., 10.4 cm)			
	Time after spindle started to rotate, sec											
	30	60	90	120	30	60	90	120	30	60	90	120
6	4520	3120	2860	2600	5800	5600	5200	5080	1260	1260	1200	1200
12	1750	1720	1700	1660	3050	2750	2400	1960	960	950	940	890
30	1052	1052	960	920	1120	1060	1020	960	856	816	788	764
60	720	694	662	612	744	740	706	646	1112	952	830	726

[a]Temperature, 86° ±1.0° F.

[b]Number 3 spindle.

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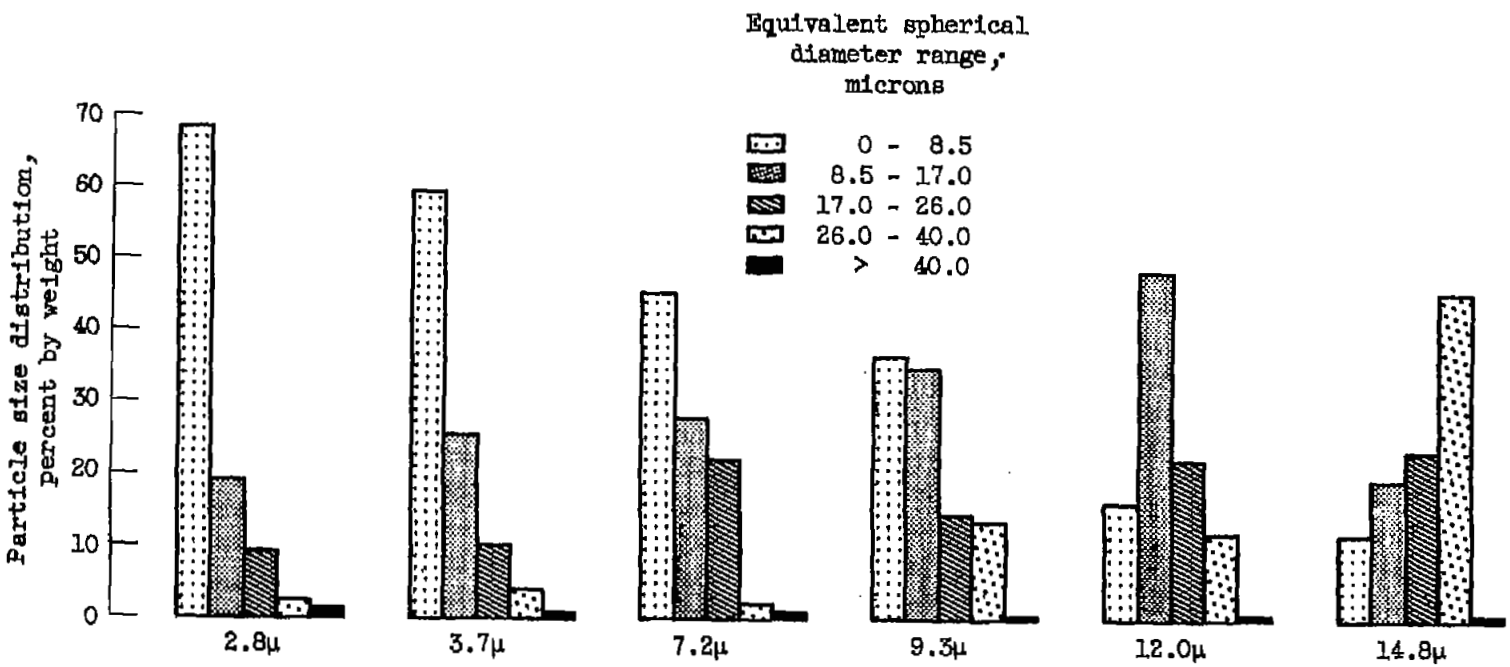

Figure 1. - Particle size distribution of magnesium powder according to Roller analyzer. (The number below each bar graph is the average equivalent particle diameter in microns according to the Fisher Sub-Sieve Sizer.)

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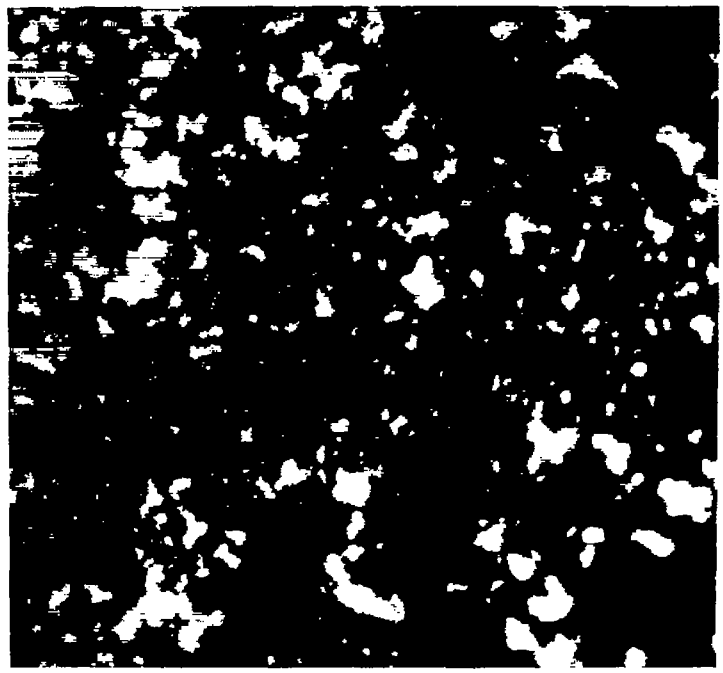
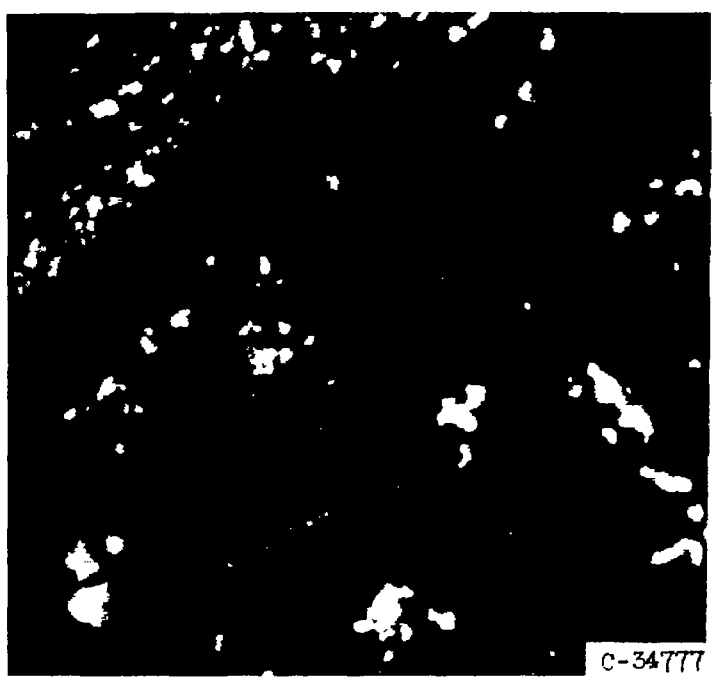


(a) Average equivalent spherical diameter, 2.8 microns.



(b) Average equivalent spherical diameter, 3.7 microns.

Figure 2. - Photomicrographs of atomized magnesium particles (X400).

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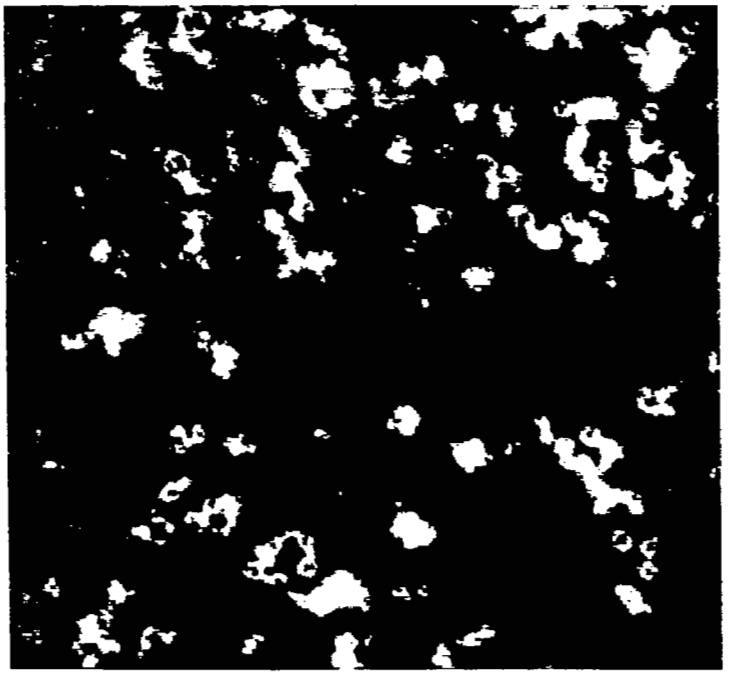
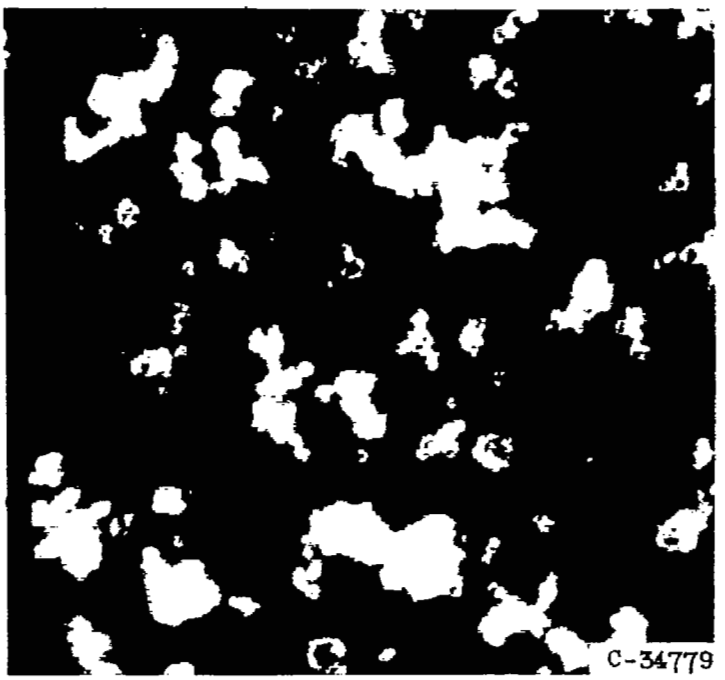
CO-4



(c) Average equivalent spherical diameter, 7.2 microns.



(d) Average equivalent spherical diameter, 9.3 microns.

Figure 2. - Continued. Photomicrographs of atomized magnesium particles (X400).

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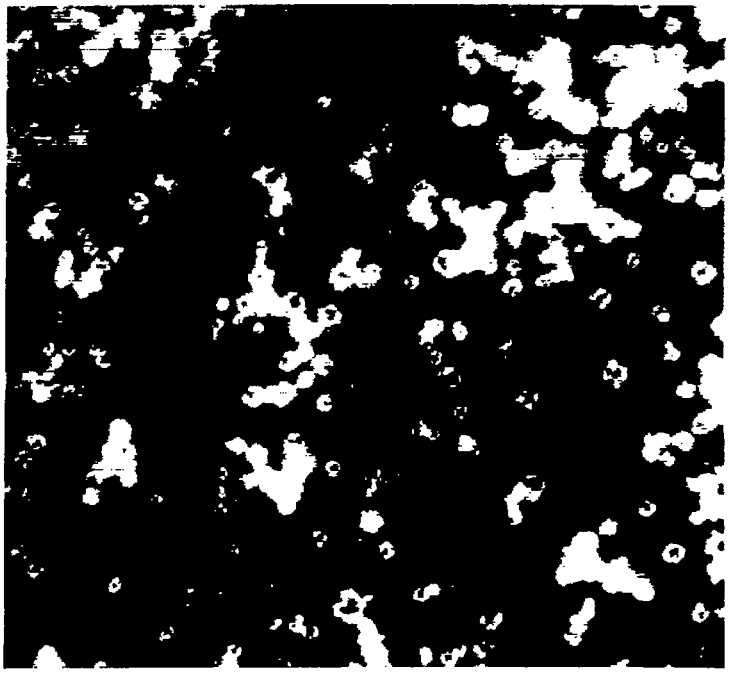
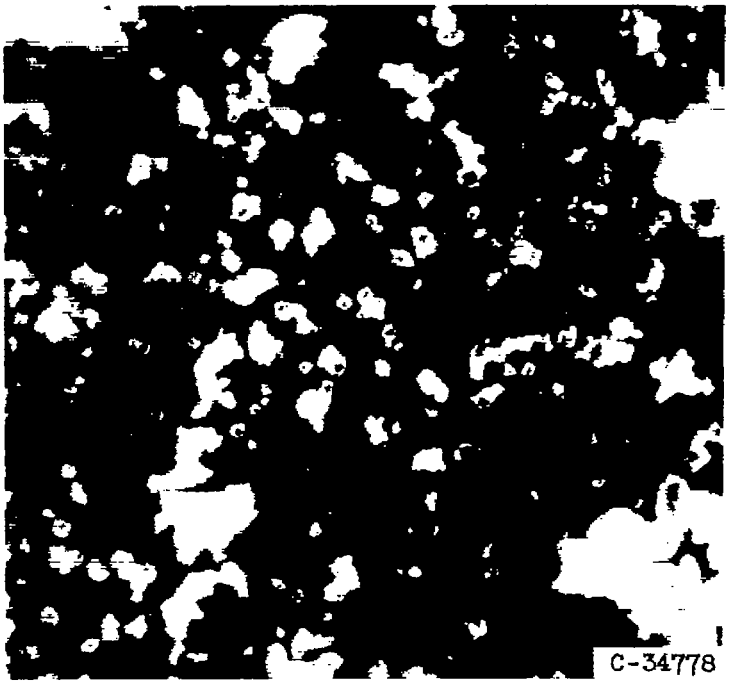


(e) Average equivalent spherical diameter, 12.0 microns.



(f) Average equivalent spherical diameter, 14.8 microns.

Figure 2. - Concluded. Photomicrographs of atomized magnesium particles (X400).

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CO-4 back

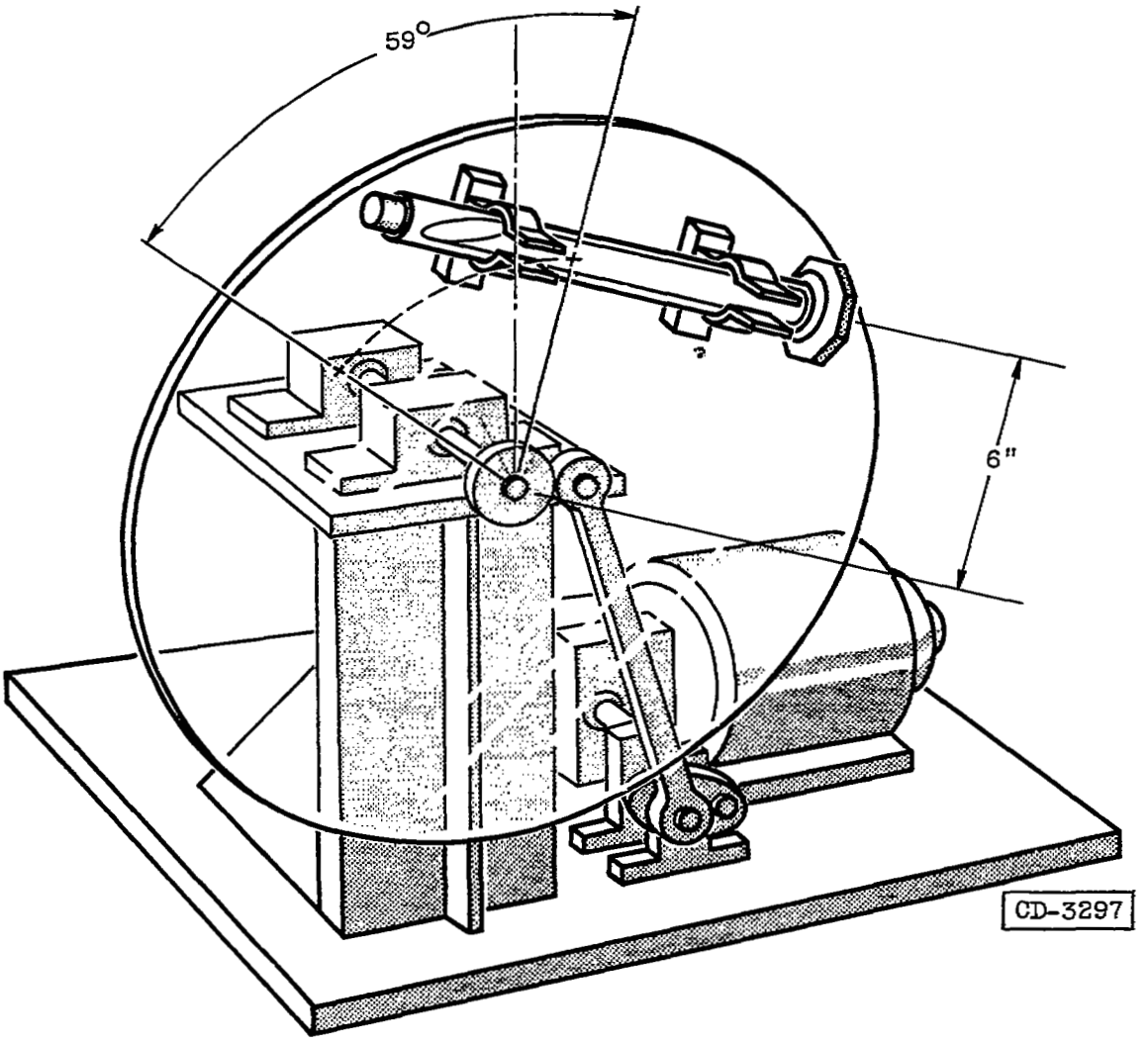

Figure 3. - Shaker used in determination of redispersibility.

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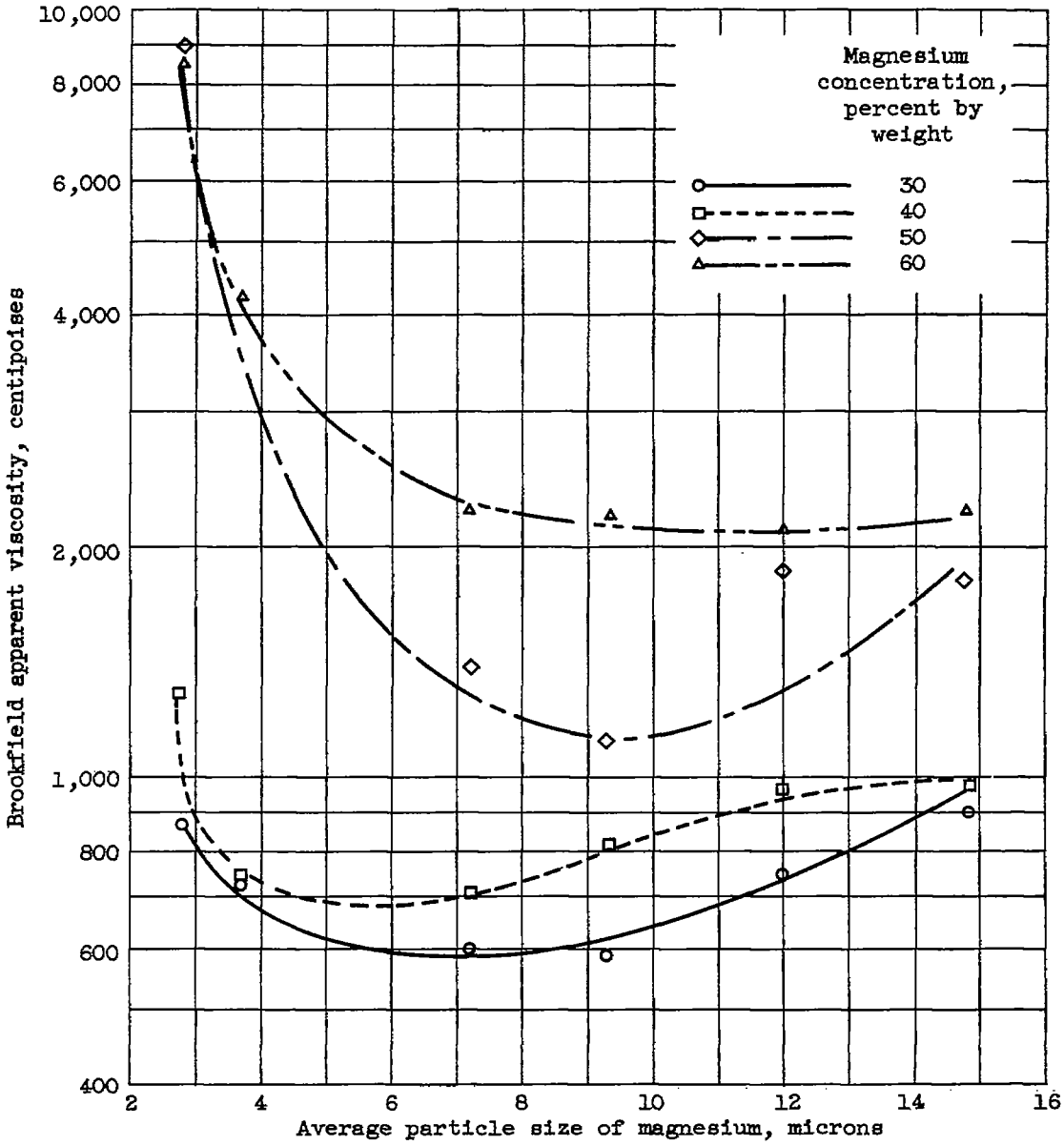
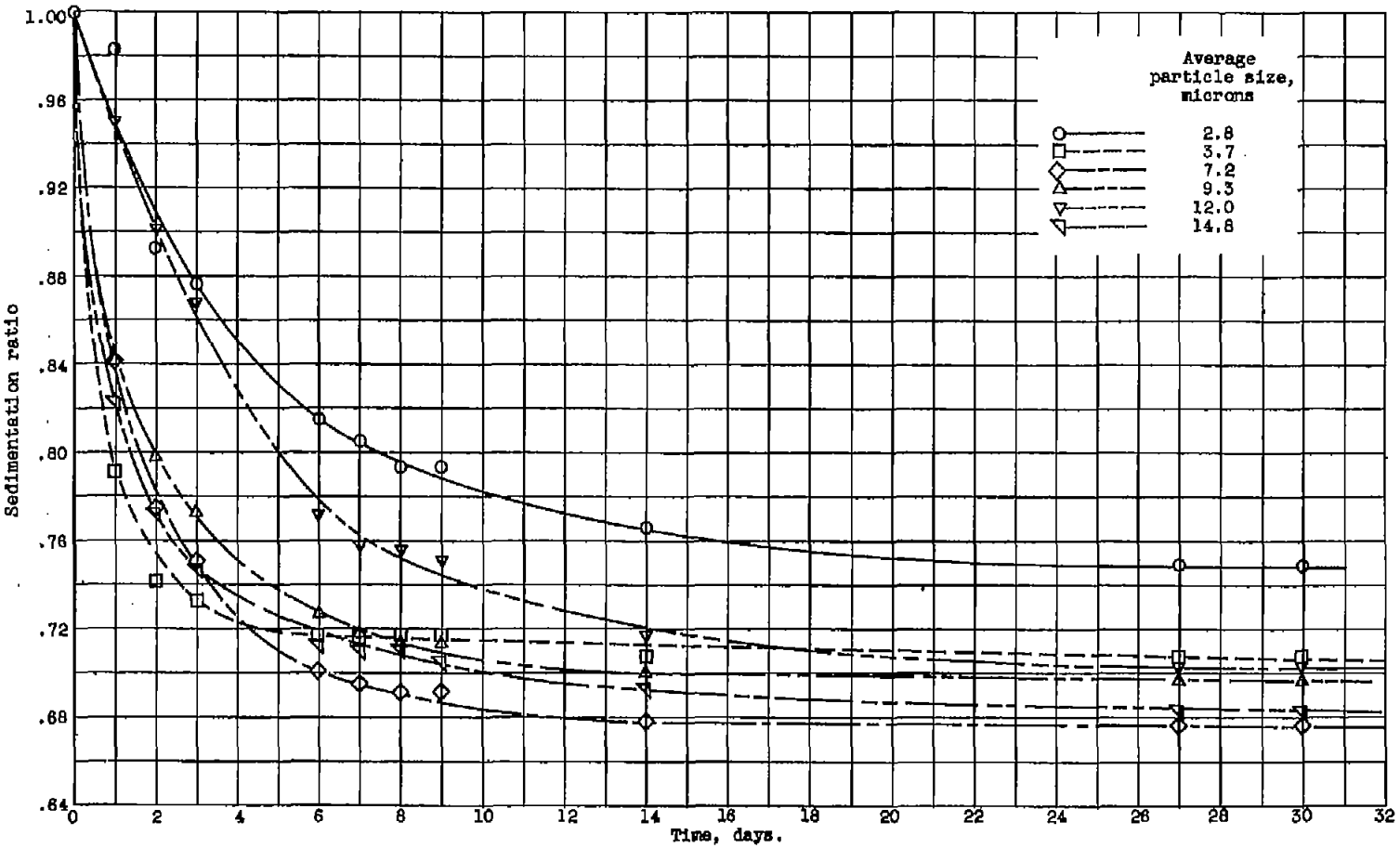
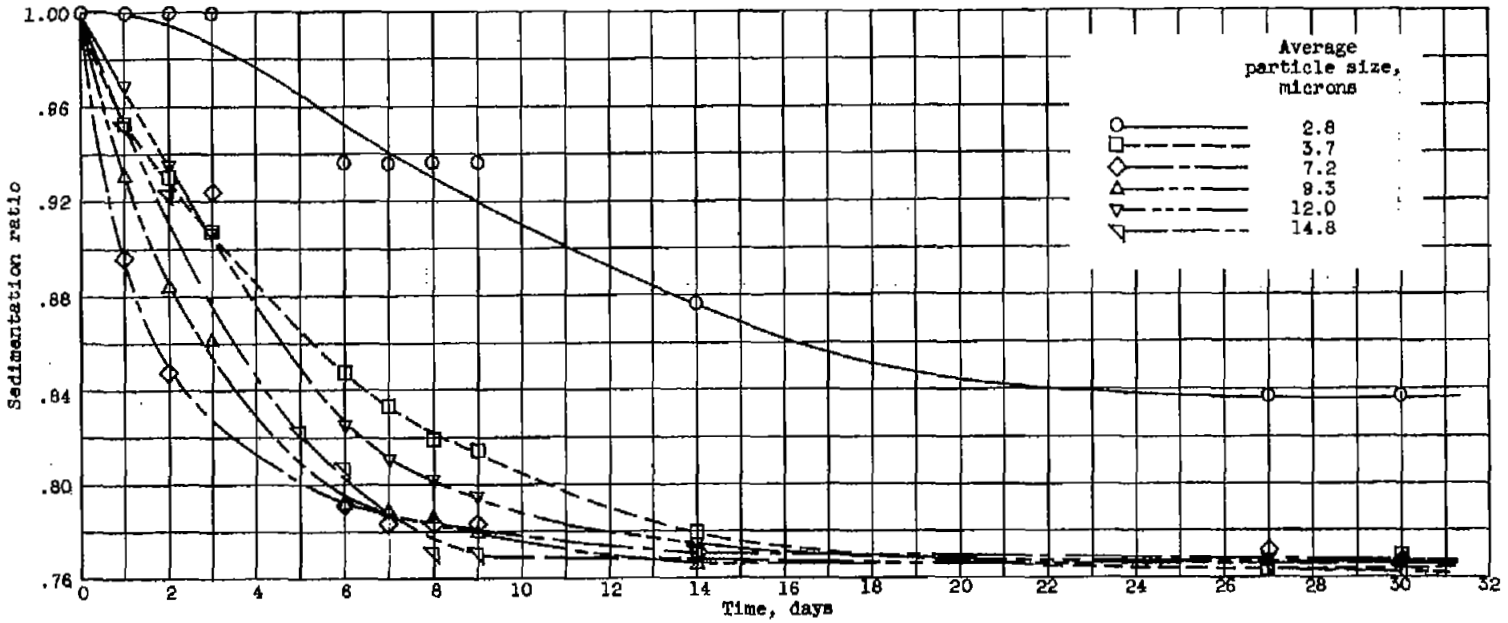


Figure 4. - Effect of average particle size on viscosity of petrolatum-stabilized magnesium slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4 by weight. Temperature, 86° ± 1.0° F.



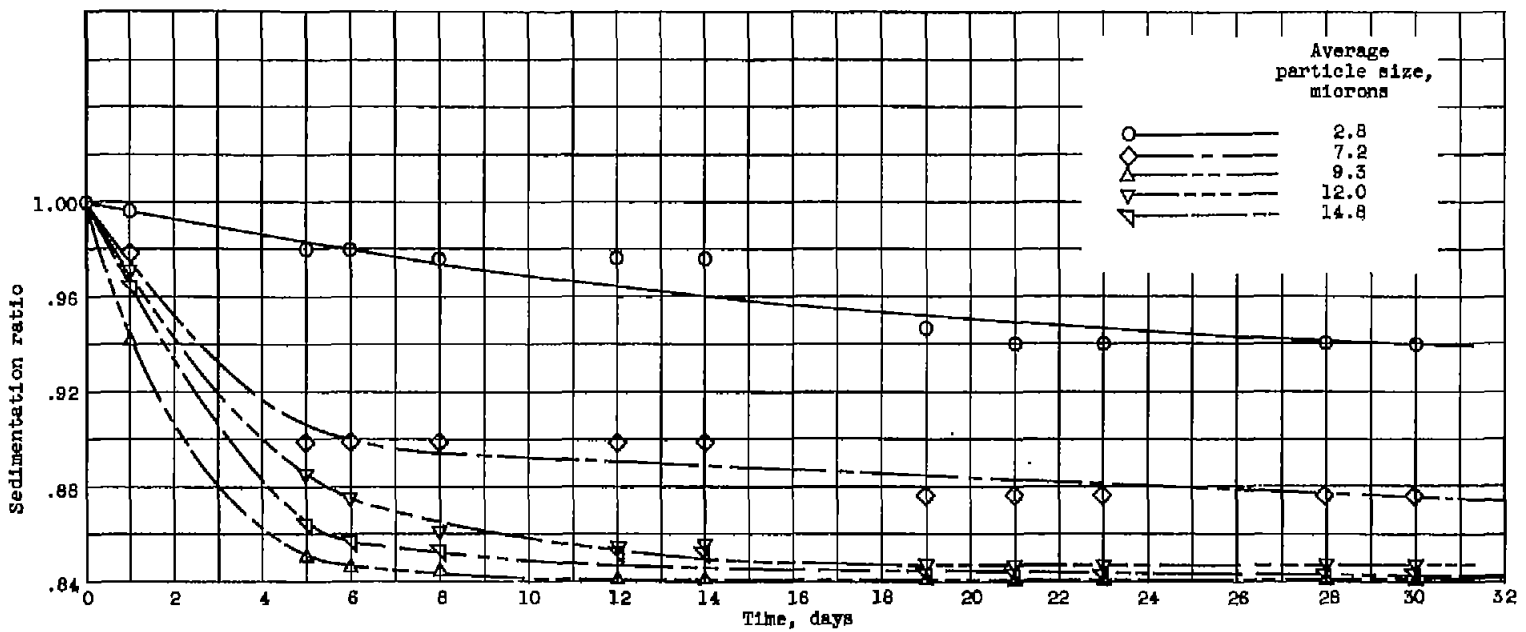
(a) Magnesium, 30 percent by weight.

Figure 5. - Effect of time on sedimentation ratio of petrolatum-stabilized slurries containing magnesium of various average particle sizes. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.



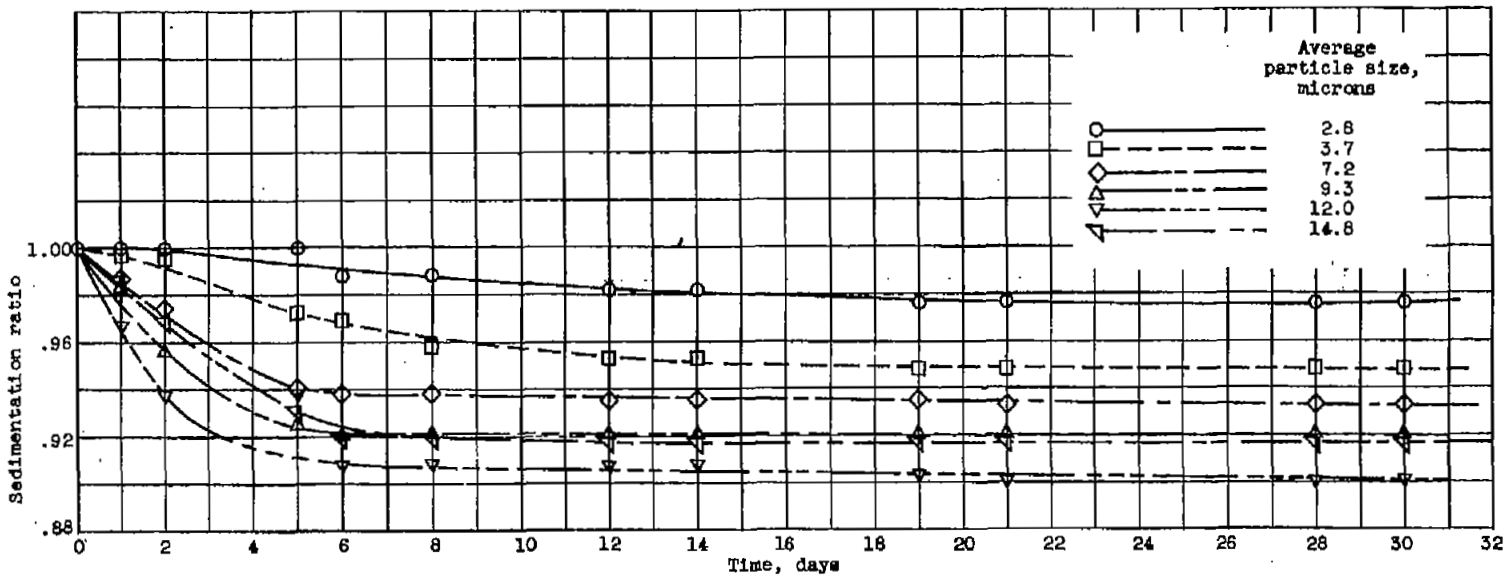
(b) Magnesium, 40 percent by weight.

Figure 5. - Continued. Effect of time on sedimentation ratio of petrolatum-stabilized slurries containing magnesium of various average particle sizes. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.



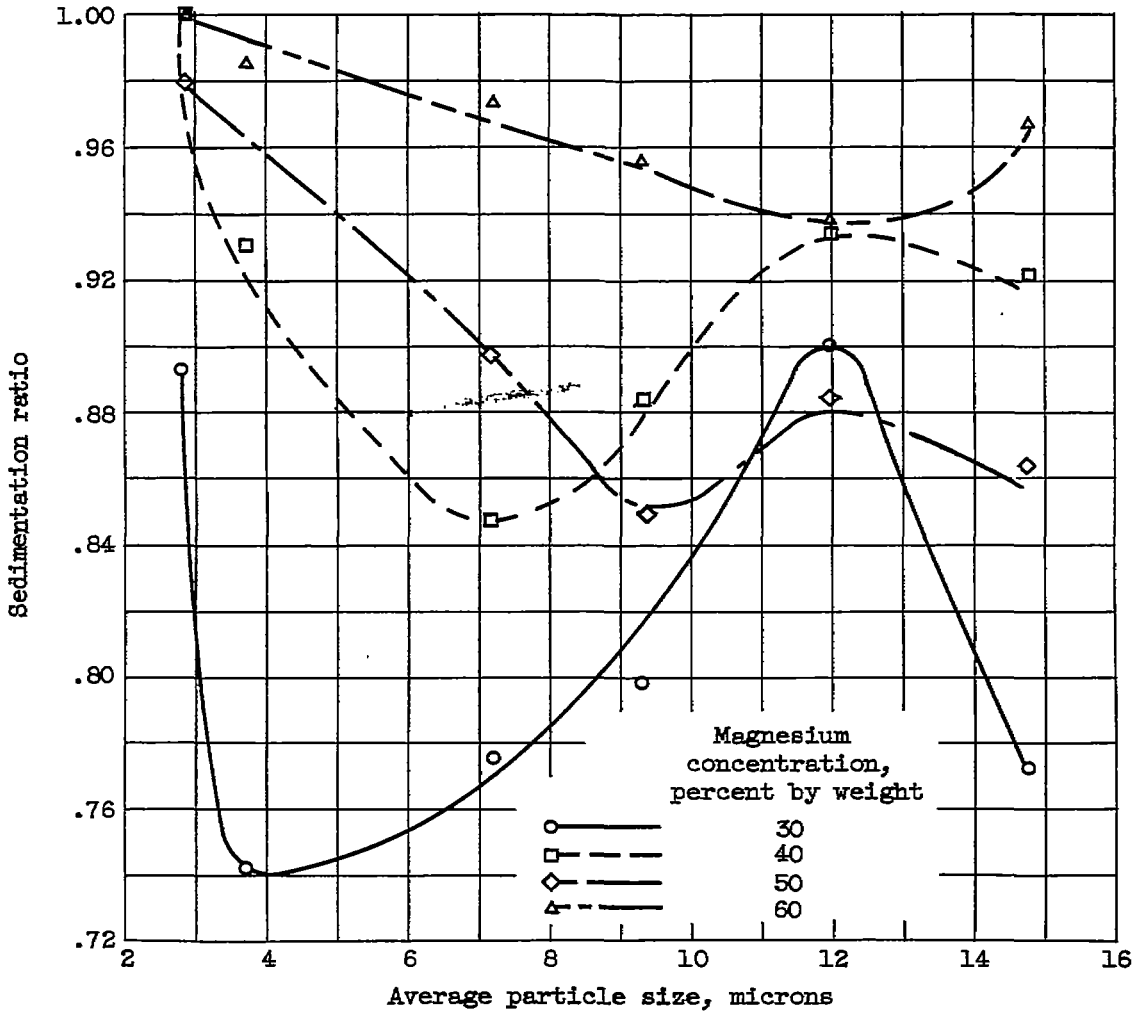
(c) Magnesium, 50 percent by weight.

Figure 5. - Continued. Effect of time on sedimentation ratio of petrolatum-stabilized slurries containing magnesium of various average particle sizes. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.



(d) Magnesium, 60 percent by weight.

Figure 5. - Concluded. Effect of time on sedimentation ratio of petrolatum-stabilized slurries containing magnesium of various average particle sizes. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.



(a) Time, 2 days.

Figure 6. - Effect of average particle size on sedimentation ratio of petrolatum-stabilized magnesium slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.

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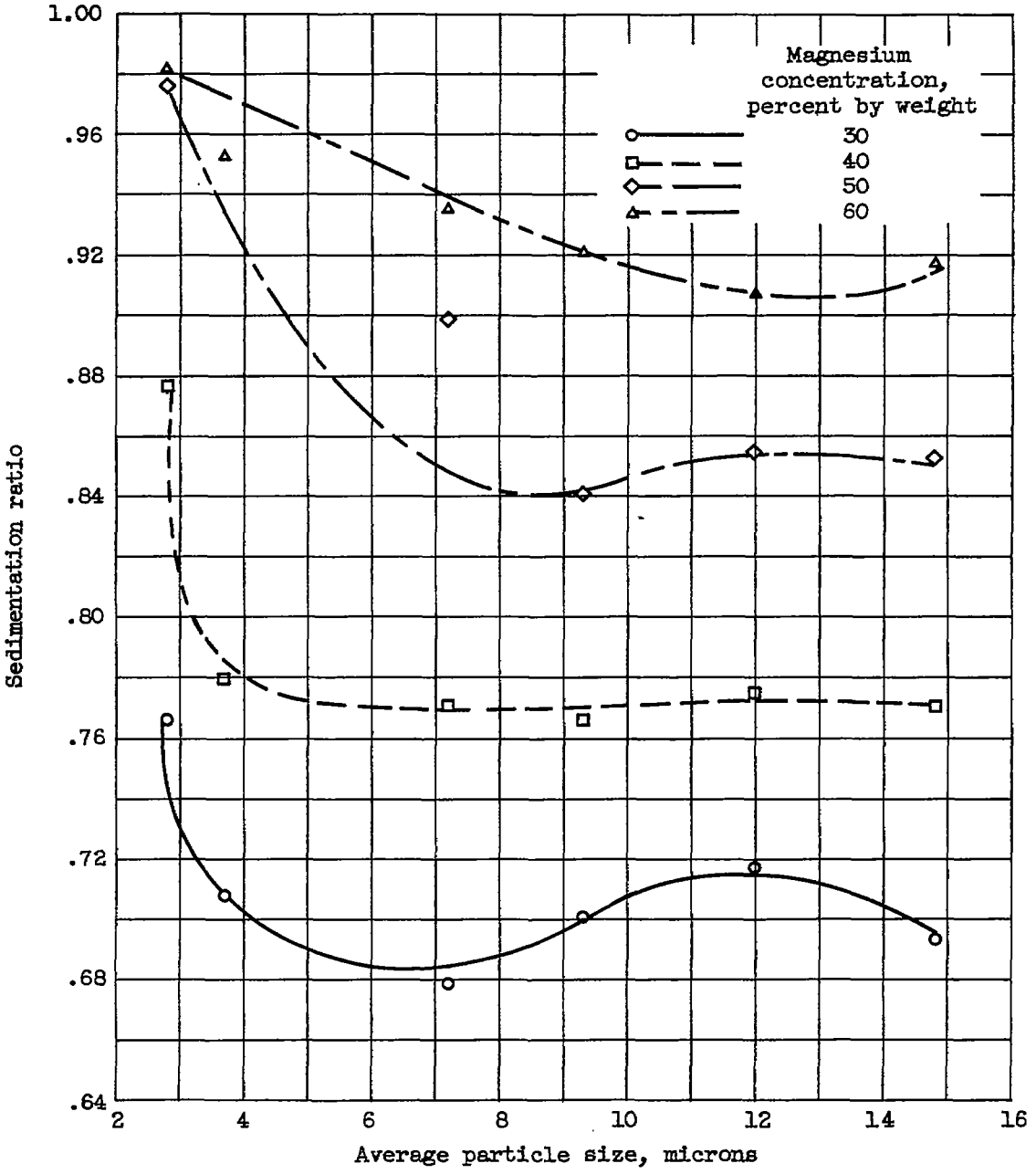

(b) Time, 14 days.

Figure 6. - Concluded. Effect of average particle size on sedimentation ratio of petrolatum-stabilized magnesium slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight.

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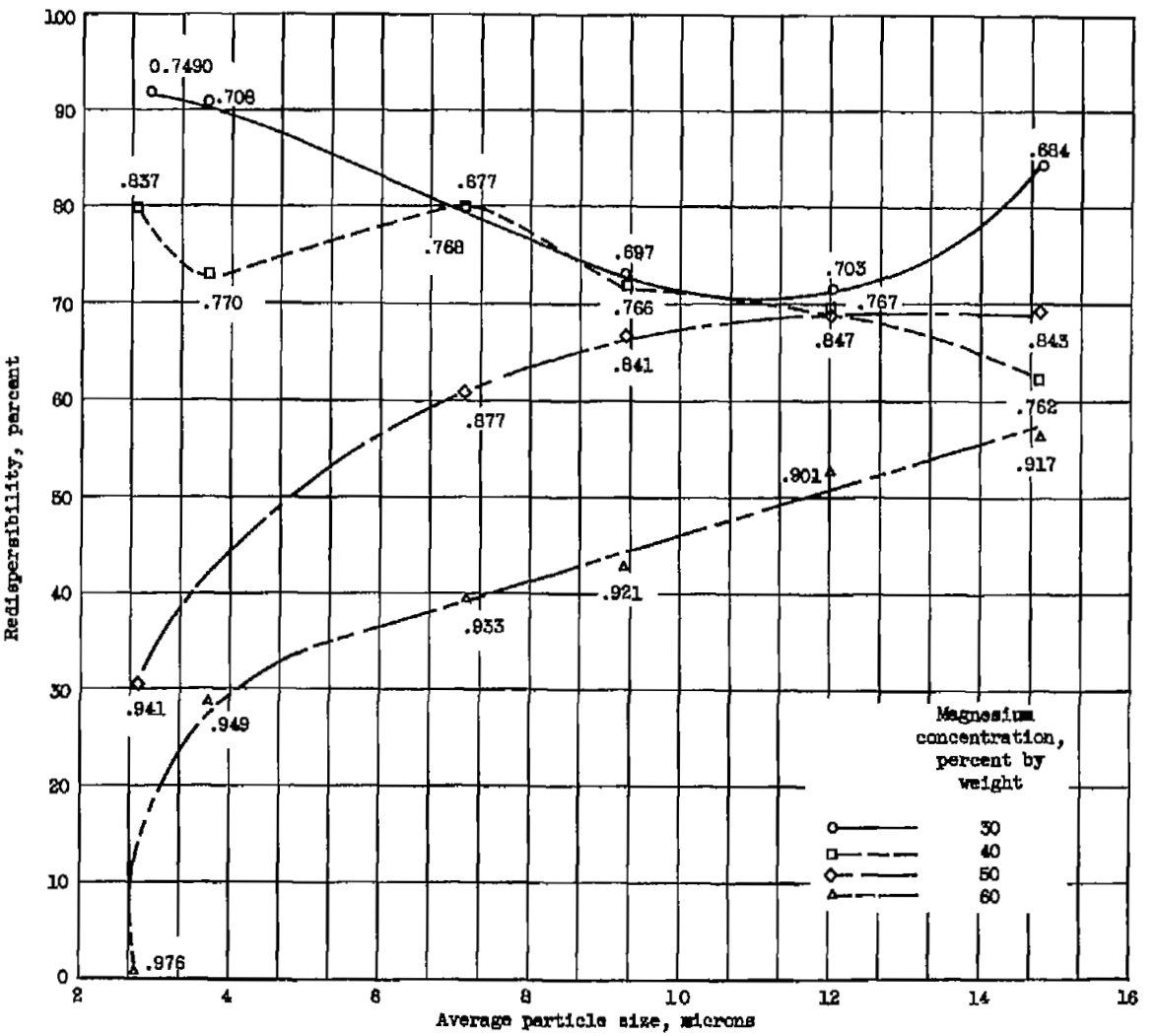

Figure 7. - Effect of average particle size of redispersibility of petrolatum-stabilized magnesium slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight. (The number beside each data point is the sedimentation ratio at 30 days.)

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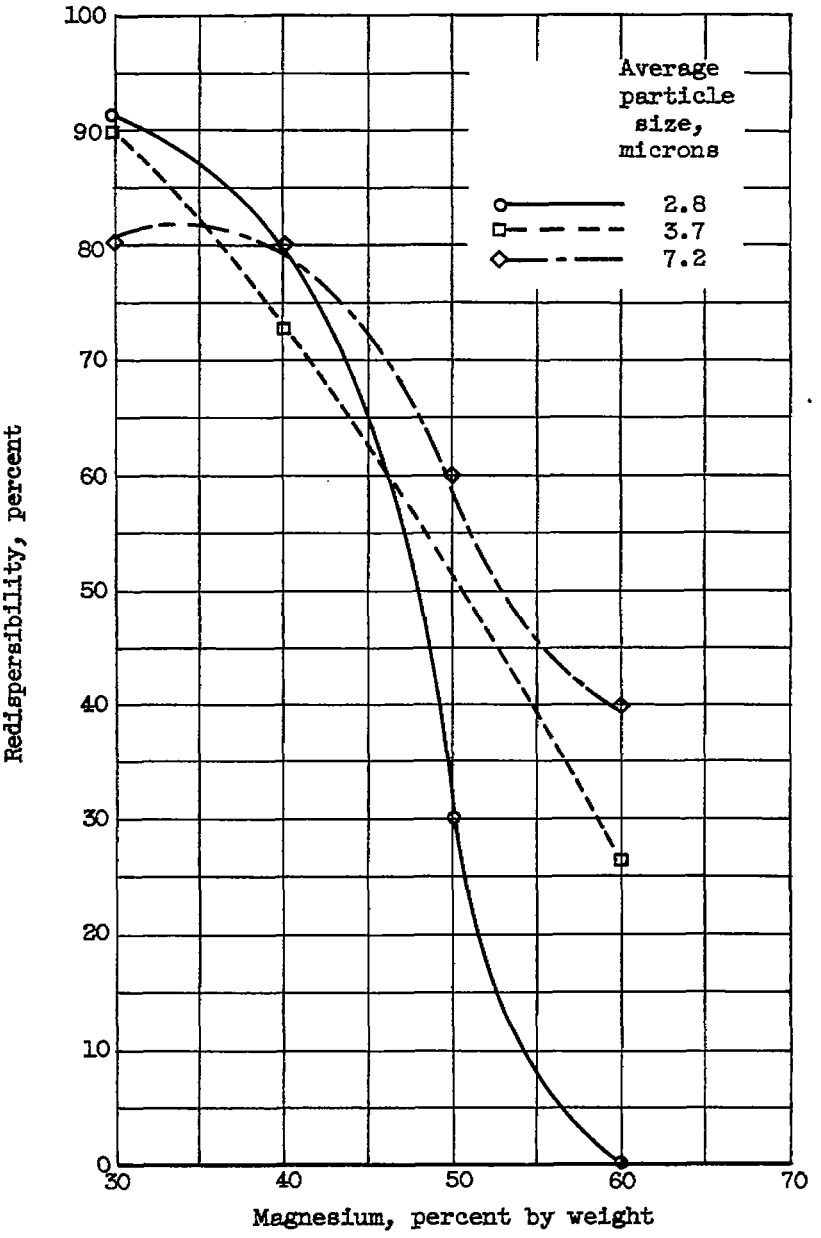

(a) Magnesium particle sizes, 2.8, 3.7, and 7.2 microns.

Figure 8. - Effect of concentration of various particle sizes of magnesium on redispersibility of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; sedimentation ratio at 30 days.

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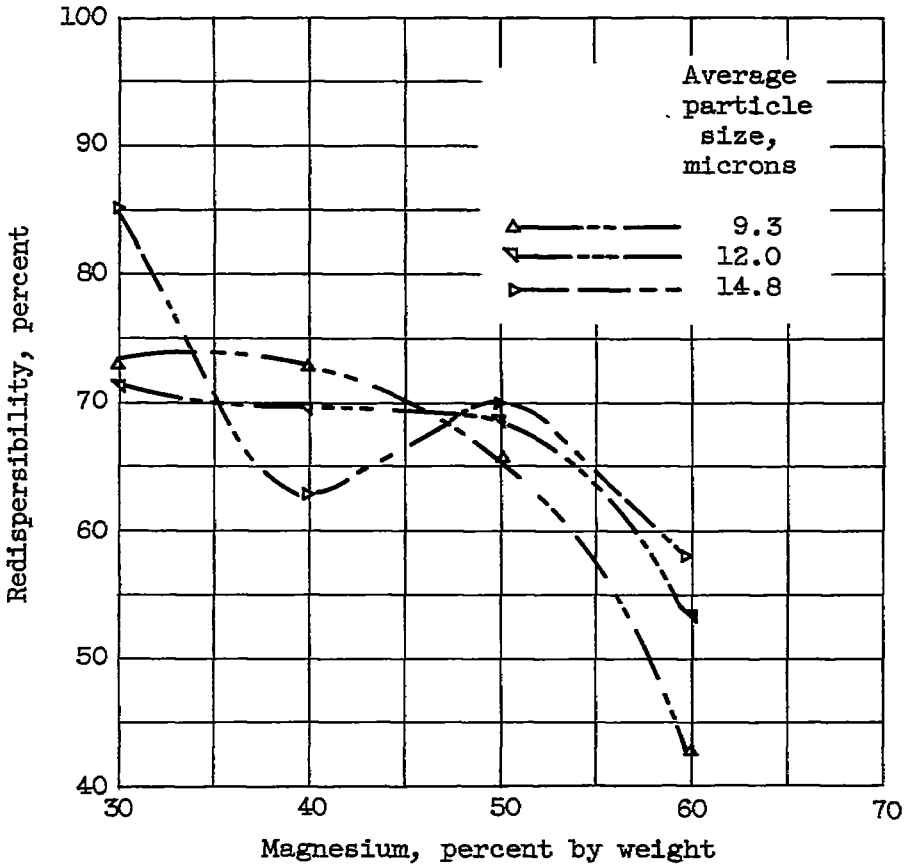
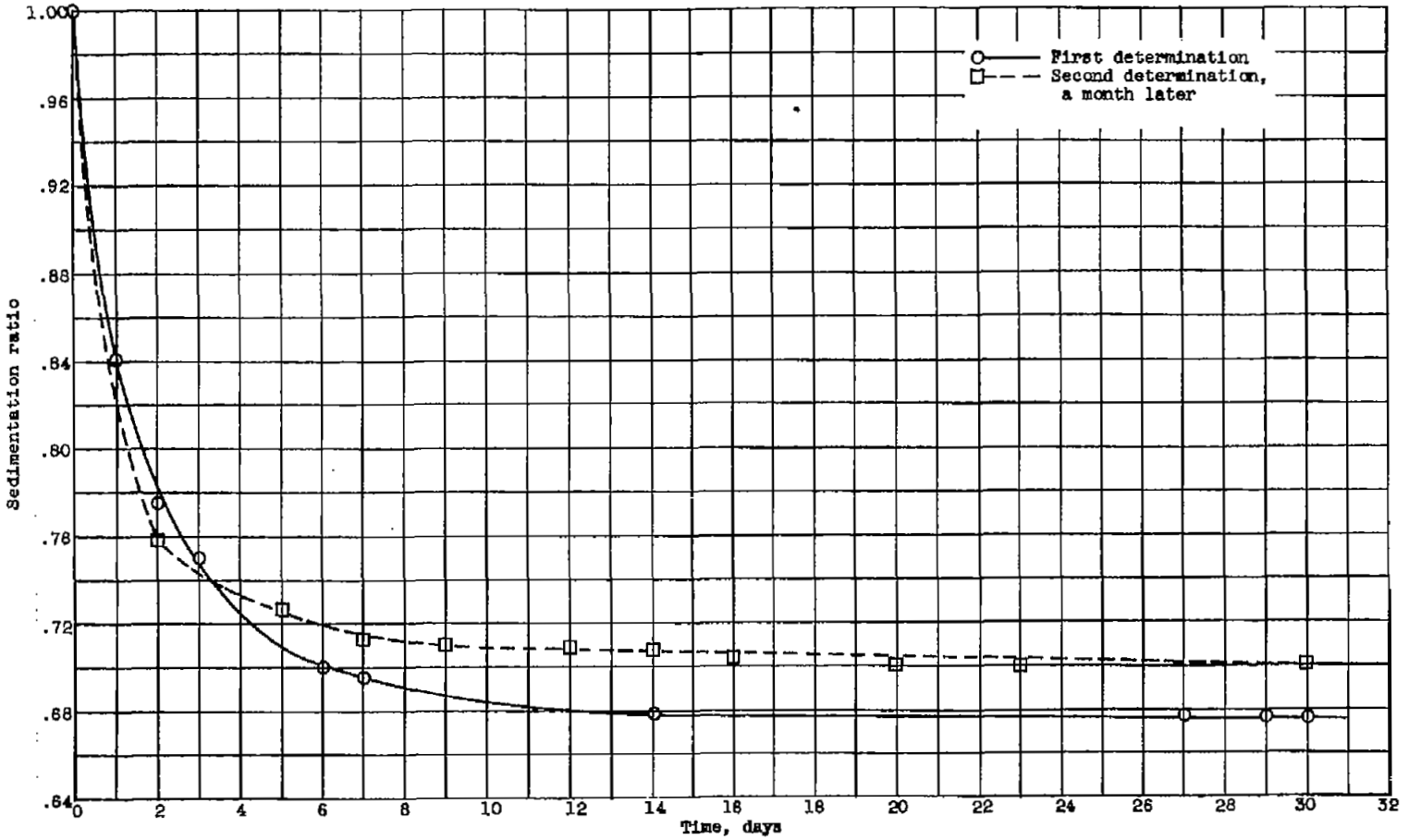


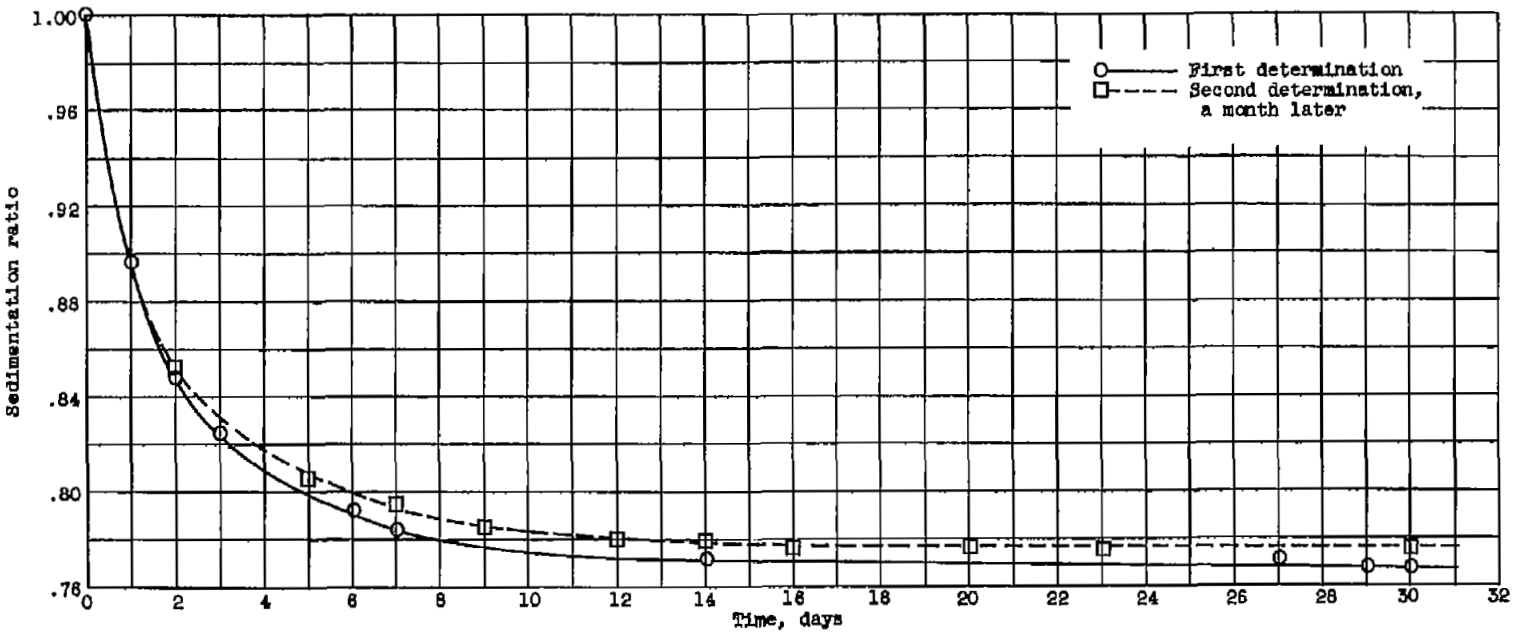
(b) Magnesium particle sizes, 9.3, 12.0, and 14.8 microns.

Figure 8. - Concluded. Effect of concentration of various particle sizes of magnesium on redispersibility of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; sedimentation ratio at 30 days.



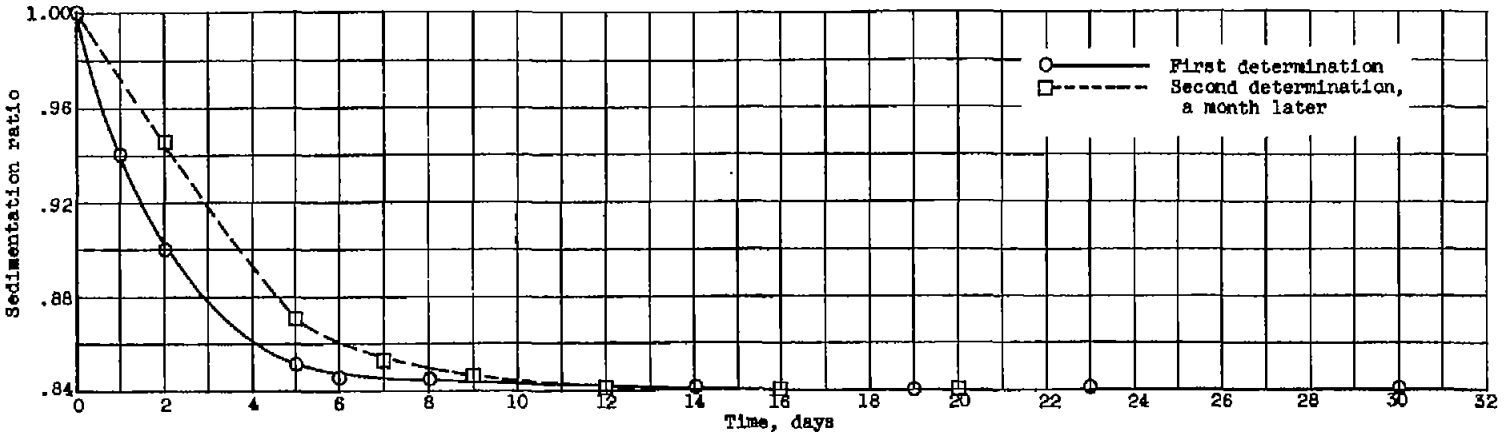
(a) Magnesium, 30 percent by weight.

Figure 9. - Reproducibility of sedimentation ratio - time relation of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns.



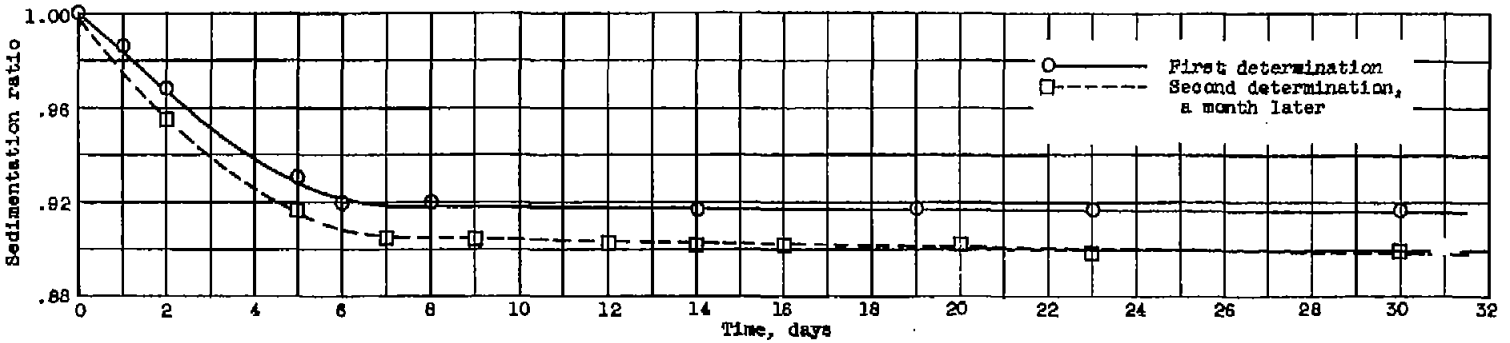
(b) Magnesium, 40 percent by weight.

Figure 9. - Continued. Reproducibility of sedimentation ratio - time relation of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns.



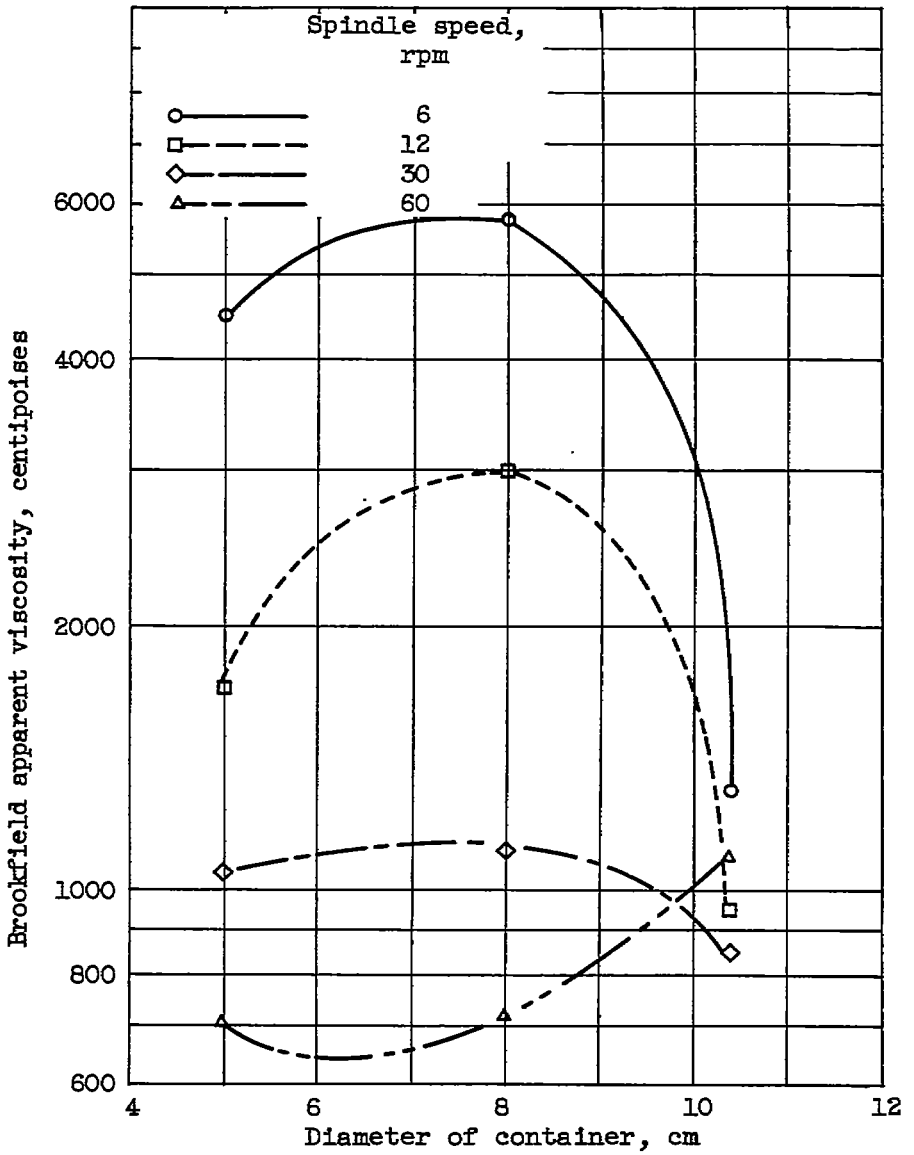
(c) Magnesium, 50 percent by weight.

Figure 9. - Continued. Reproducibility of sedimentation ratio - time relation of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns.



(d) Magnesium, 60 percent by weight.

Figure 9. - Concluded. Reproducibility of sedimentation ratio - time relation of petrolatum-stabilized magnesium - JP-4 slurries. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns.



(a) Viscosity readings taken 30 seconds after spindle started to rotate.

Figure 10. - Effect of container size on viscosity of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position on baffle and spindle, constant; spindle size, number 3; temperature, 86° ± 1.0° F.

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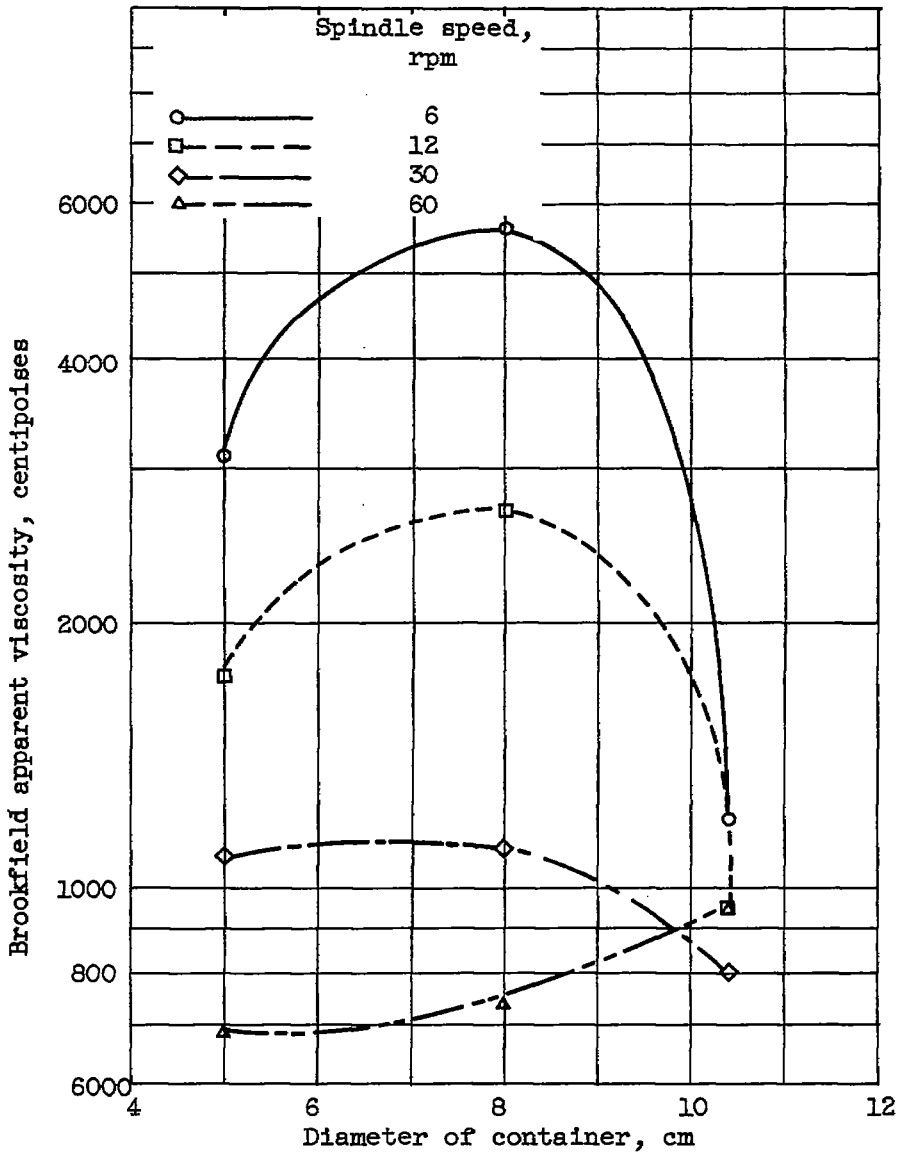

(b) Viscosity readings taken 60 seconds after spindle started to rotate.

Figure 10. - Continued. Effect of container size on viscosity of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of baffle and spindle, constant; spindle size, number 3; temperature, 86° ± 1.0° F.

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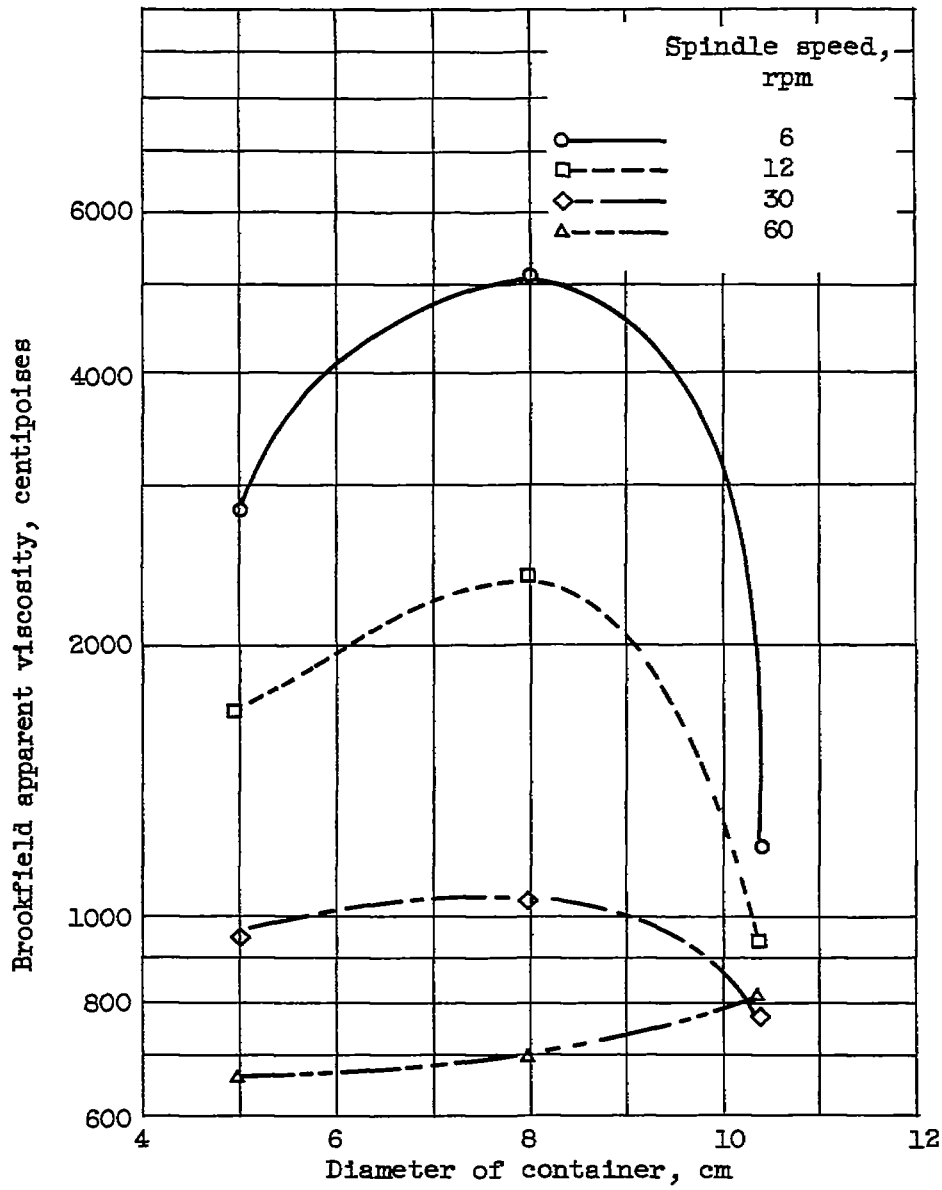

(c) Viscosity readings taken 90 seconds after spindle started to rotate.

Figure 10. - Continued. Effect of container size on viscosity of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of baffle and spindle, constant; spindle size, number 3; temperature, 86° ± 1.0° F.

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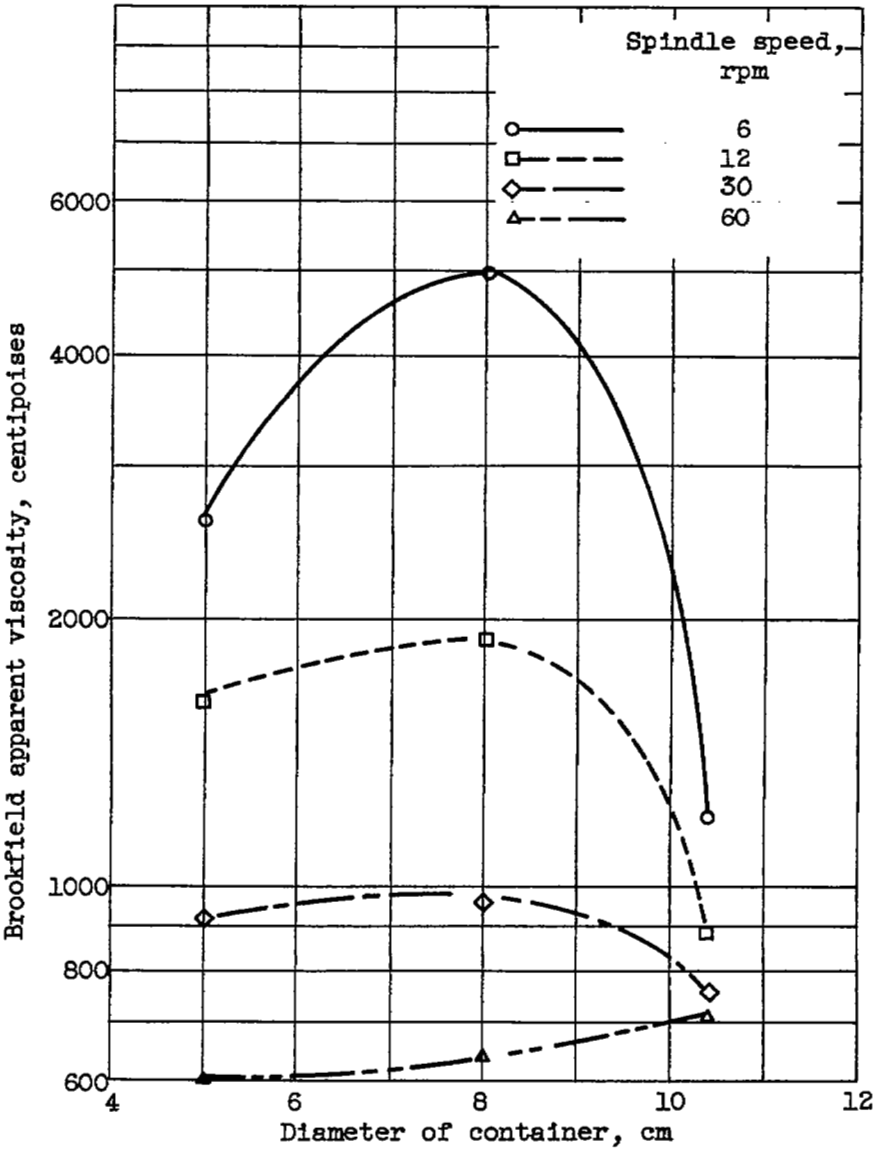

(d) Viscosity readings taken 120 seconds after spindle started to rotate.

Figure 10. - Concluded. Effect of container size on viscosity of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of baffle and spindle, constant; spindle size, number 3; temperature, 86° ± 1.0° F.

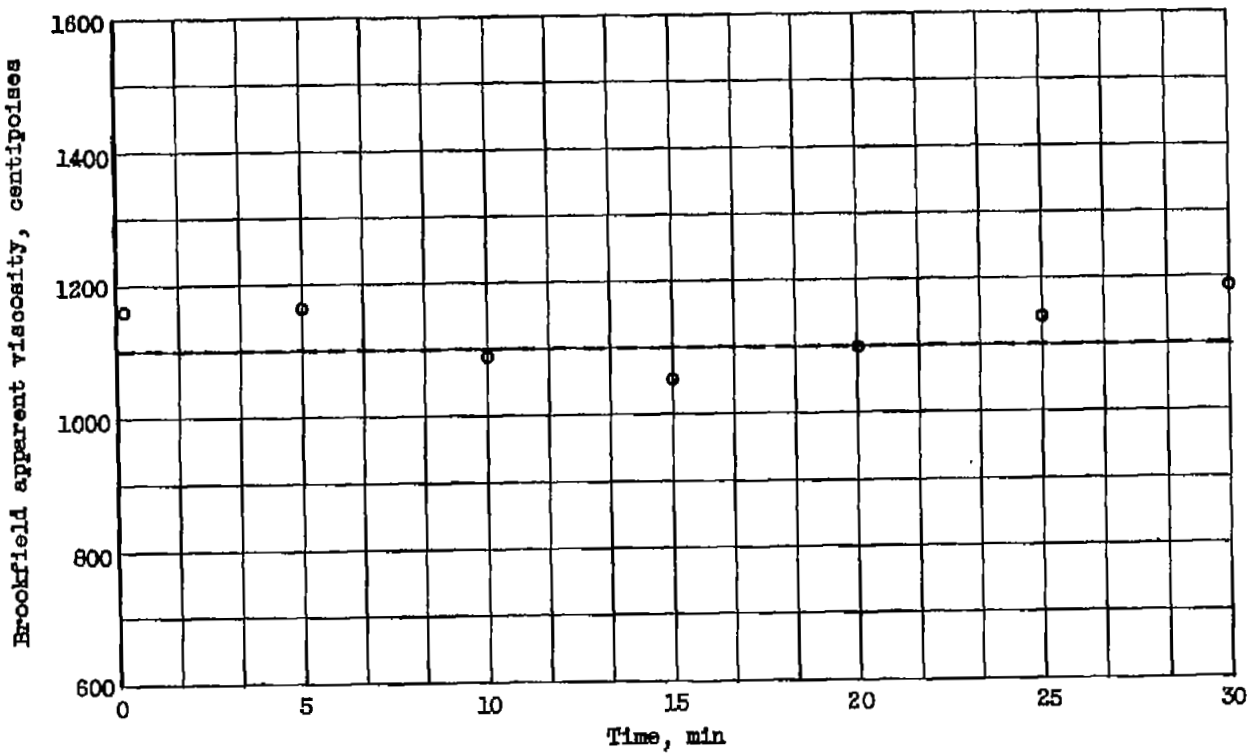

Figure 11. - Effect of time on viscosity measurements of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of baffle and spindle, constant; spindle size, number 3; spindle speed, 12 revolutions per minute; container size, 1 quart; container inside diameter, 10.4 centimeters; time, 30 seconds after spindle started to rotate; temperature, 86° ± 1.0° F; maximum deviation, 10 percent.

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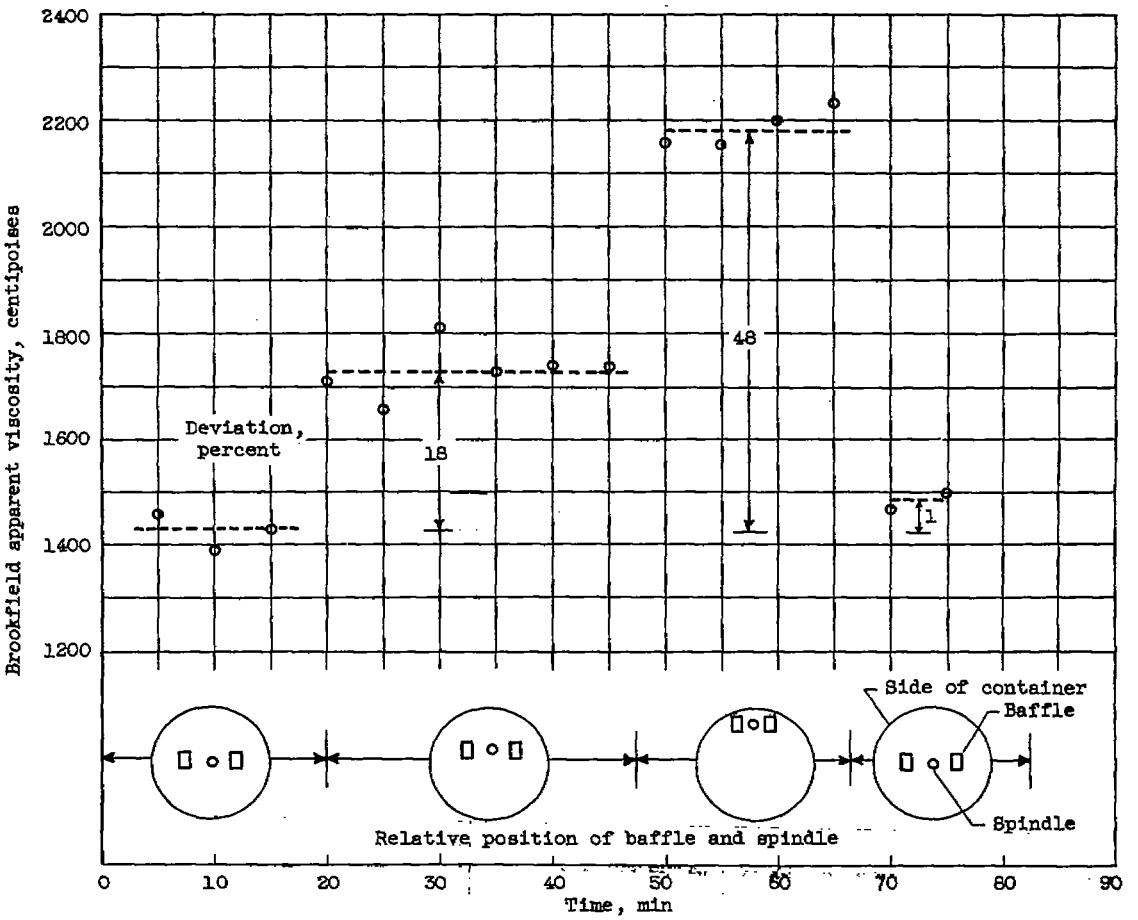
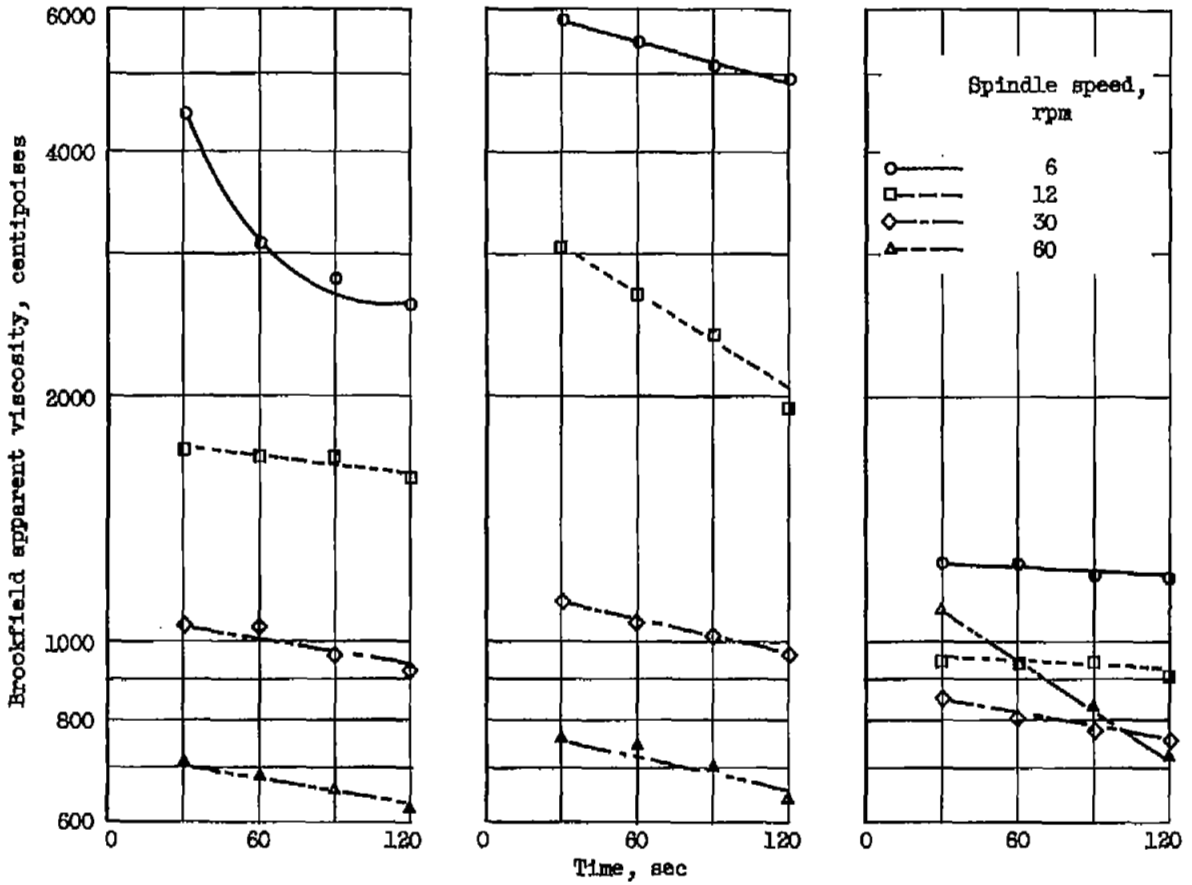


Figure 12. - Effect of time on viscosity measurements of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of baffle and spindle, variable; spindle size, number 3; spindle speed, 12 revolutions per minute; container inside diameter, 10.4 centimeters; time, 30 seconds after spindle started to rotate; temperature, 86° ± 1.0° F.

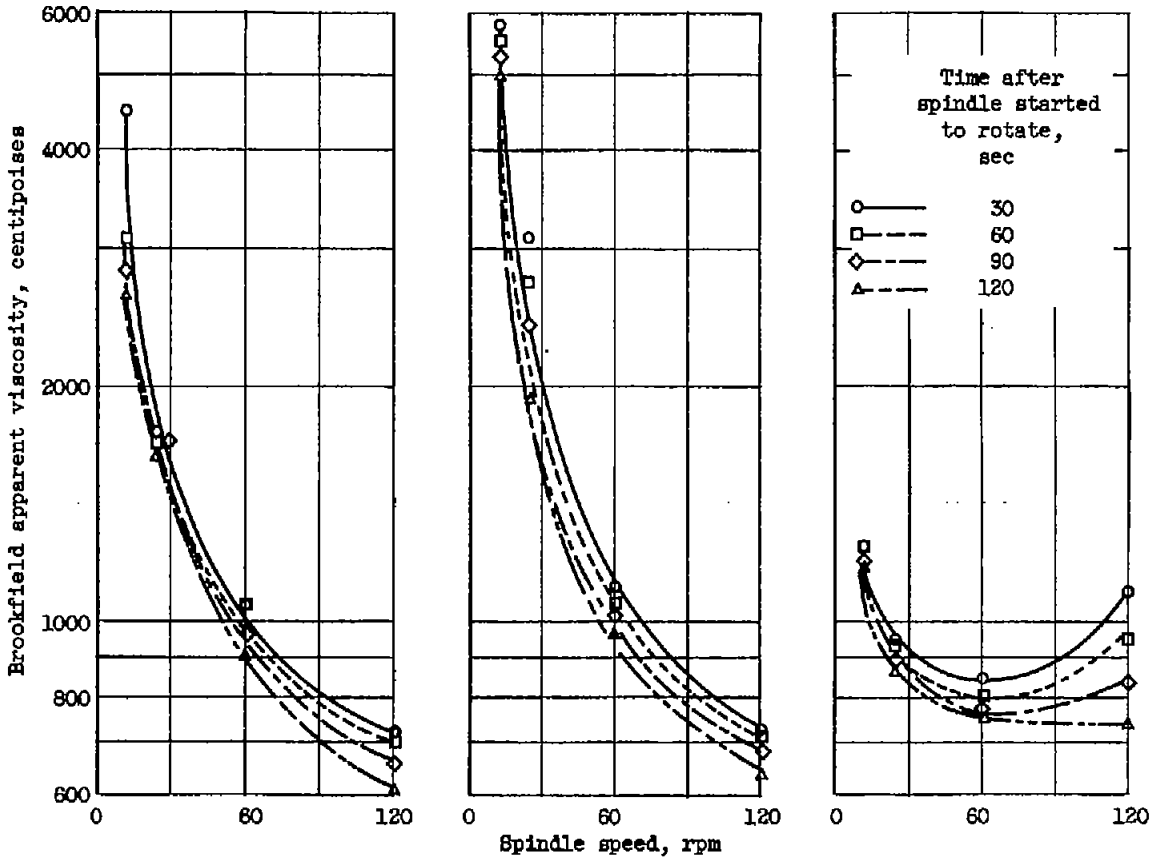


(a) Container, 100-milliliter glass beaker; inside diameter, 5 centimeters.

(b) Container, 400-milliliter glass beaker; inside diameter, 8 centimeters.

(c) Container, round, 1-quart can; inside diameter, 10.4 centimeters.

Figure 13. - Effect of time after spindle started to rotate on viscosity measurements of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; relative position of spindle and baffle, constant; spindle size, number 3; temperature, 86° ± 1.0° F.



(a) Container, 100-milliliter glass beaker; inside diameter, 5 centimeters.

(b) Container, 400-milliliter glass beaker; inside diameter, 8 centimeters.

(c) Container, round, 1-quart can; inside diameters, 10.4 centimeters.

Figure 14. - Effect of spindle speed on viscosity measurements of petrolatum-stabilized magnesium - JP-4 slurry. Hydrocarbon medium composed of 40 percent petrolatum and 60 percent MIL-F-5624A, grade JP-4, by weight; magnesium particle size, 7.2 microns; position of spindle and baffle, constant; spindle size, number 3; temperature, $86^{\circ} \pm 1.0^{\circ}$ F.

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